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NASA CONTRACTOR  
REPORT

NASA CR 144339

(NASA-CR-144339) INVESTIGATION OF LOW COST,  
HIGH RELIABILITY SEALING TECHNIQUES FCR  
HYBRID MICROCIRCUITS, PHASE 1 Final Report  
(Rockwell International Corp., Anaheim,  
Calif.) 67 p HC \$4.50

N76-27364

Unclassified  
CSCL 11A G3/24 44614

INVESTIGATION OF LOW COST, HIGH RELIABILITY SEALING  
TECHNIQUES FOR HYBRID MICROCIRCUITS

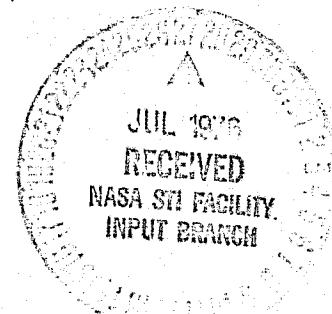
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April 1976

Phase I, Final Report

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Prepared for

NASA - GEORGE C. MARSHALL SPACE FLIGHT CENTER  
Marshall Space Flight Center, Alabama 35812

## TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO. NASA CR-144339	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.	
4. TITLE AND SUBTITLE Investigation of Low Cost, High Reliability Sealing Techniques for Hybrid Microcircuits		5. REPORT DATE April 1976	
7. AUTHOR(S) K. L. Perkins and J. J. Licari		6. PERFORMING ORGANIZATION CODE	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Rockwell International - Autonetics Group Post Office Box 3105 Anaheim, California 92803		8. PERFORMING ORGANIZATION REPORT # C75-588/201	
12. SPONSORING AGENCY NAME AND ADDRESS National Aeronautics and Space Administration Washington, D. C. 20546		10. WORK UNIT NO.	
		11. CONTRACT OR GRANT NO. NAS8-31517	
		13. TYPE OF REPORT & PERIOD COVERED Contractor Report Phase I, Final	
		14. SPONSORING AGENCY CODE	
15. SUPPLEMENTARY NOTES			
16. ABSTRACT <p>A preliminary investigation was made to determine the feasibility of using adhesive package sealing for hybrid microcircuits for NASA/MSFC applications. Major effort consisted of (1) surveying representative hybrid manufacturers to assess the current use of adhesives for package sealing; (2) making a cost comparison of metallurgical versus adhesive package sealing; (3) determining the seal integrity of gold-plated Kovar flatpack-type packages sealed with selected adhesives after they had been subjected to MIL-STD-883A, Class A, thermal shock, temperature cycling, mechanical shock, and constant acceleration test environments; and, (4) defining a more comprehensive study to continue the evaluation of adhesives for package sealing.</p>			
<p>Results showed that 1.27 cm square gold-plated Kovar flatpack-type packages sealed with the film adhesives Ablefilm 507, 529, and 550 and the paste adhesive Epo-Tek H77 retained their seal integrity after all tests, and that similarly prepared 2.54 cm square packages retained their seal integrity after all tests except the 10,000 g's constant acceleration test. These results are encouraging, but by no means sufficient to establish the suitability of adhesives for sealing high reliability hybrid microcircuits. Much remains to be done to determine the degree to which adhesives are suitable for this application, and to establish an adequate data base for writing a guideline specification for the selection and qualification of adhesives if this is justified.</p>			
17. KEY WORDS		18. DISTRIBUTION STATEMENT Publicly Available <i>J. Brooks Moore</i> F. BROOKS MOORE Director, Electronics and Control Laboratory	
19. SECURITY CLASSIF. (of this report) Uncl	20. SECURITY CLASSIF. (of this page) Uncl	21. NO. OF PAGES 64	22. PRICE NTIS

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## 1.0 INTRODUCTION

## 1.1 STUDY BACKGROUND

At the present time, only metallurgical methods are qualified for sealing hybrid microcircuit packages to be used for space and military applications. These methods include various soldering, brazing and welding techniques. While each is accompanied by its own detailed problems, with sufficient controls, all can produce hermetic seals with reasonably high yields. In general, these methods provide highly reliable hermetic seals and excellent protection for the hybrid circuits, but are also accompanied by inherent disadvantages or problems which can adversely affect the reliability of the circuits.

Depending on the method used, some limitations are:

- ° Metal particles generated during the sealing process can become entrapped in the packages and ultimately cause circuit malfunctions.
- ° Flux used in the sealing processes using Pb/Sn solder can flow into the packages and contaminate the circuits, causing corrosion and consequent degradation of performance.
- ° The high sealing temperatures (greater than 250°C) required for some of the methods, if not localized to the seal area, can adversely affect the hybrid microcircuits either by causing deterioration of wire bonds or degradation of the characteristics of electronic components.
- ° Delidding metallurgically sealed packages for rework without further damaging or contaminating the hybrid microcircuits is extremely difficult.

No existing methods of sealing hybrid packages are completely satisfactory. Related problems and/or disadvantages of the various methods range from the introduction of deleterious contaminants during the sealing process to the inability of delidding the packages for circuit rework. Consequently, there is a need to investigate and if possible develop alternate sealing methods.

One method receiving considerable attention which would obviate most of the problems mentioned is epoxy adhesive sealing. Sealing with epoxy adhesives is a low temperature process (about 150°C), does not require use of flux, eliminates metal particle contamination from the lid sealing process, and permits delidding of packages for rework. In addition, if epoxy sealing can be shown to meet NASA and military requirements, considerable cost savings can result. The qualification of epoxies for sealing would allow the use of inexpensive all-ceramic integral lead packages in lieu of currently used gold-plated Kovar packages. The use of epoxy adhesives for sealing packages, however, has been a subject of controversy. Conflicting reports exist as to whether epoxy seals can ever be used in high reliability systems. Calculations based on the known moisture permeabilities of epoxies show that a complete exchange with the moisture ambient outside the package will occur in a matter of days. On the other hand, there are reports and data showing that epoxy sealed packages that have been out in the field for over six years contain very little moisture (<1000 ppm).

A study was therefore initiated to evaluate the suitability of using epoxy adhesives to seal hybrid packages, and to develop an experimental data base upon which guidelines for epoxy sealing could be established.

## 1.2 SCOPE OF THE PRESENT STUDY

The objective of the overall study is to investigate low cost, high reliability sealing techniques for hybrid microcircuit packages. The objective of this initial portion of the study (Phase I) was to conduct a preliminary investigation of epoxy adhesives to assess their feasibility for this application. This effort consisted of the following four tasks:

Task 1 - Survey representative hybrid microcircuit manufacturers to assess the current use of epoxy adhesives for package sealing.

Task 2 - Perform a cost comparison of metallurgical versus epoxy adhesive package sealing.

Task 3 - Seal gold-plated Kovar packages with selected epoxy adhesives and determine their seal integrity after the packages are subjected to MIL-STD-883A thermal shock, temperature cycling, mechanical shock, and constant acceleration test environments.

Task 4 - On the basis of the results obtained in Task 3, define a detailed test program to complete the evaluation of epoxy adhesives for package sealing.

## 2.0 TECHNICAL DISCUSSION

## 2.1 CURRENT USE OF ADHESIVES FOR PACKAGE SEALING

2.1.1 Survey of Adhesive Manufacturers

Ablestik Laboratories and Epoxy Technology were contacted to determine which adhesives they are currently recommending for package sealing, to define appropriate procedures for using them, and to obtain a list of hybrid manufacturers who are currently using or evaluating their adhesives for package sealing. Ablestik recommended two adhesives, Ablefilm 529 and Ablefilm 550 and provided a list of ten companies using their adhesives. Ablestik feels that there is no question that adhesive sealed packages can meet the MIL-STD-883A seal test requirement and retain their seal integrity for several years. Epoxy Technology also recommended two adhesives for consideration, Epo-Tek H74 and Epo-Tek H77. Both of these are filled epoxy paste adhesives. Epo-Tek H77 is touted as particularly resistant to moisture. Epoxy Technology feels strongly that paste adhesives are superior to film adhesives for package sealing. They provided the names of two companies currently using Epo-Tek H77 for package sealing.

2.1.2 Survey of Hybrid Microcircuit Manufacturers

Twelve companies considered to be representative of hybrid manufacturers (including most of those suggested by Ablestik and Epoxy Technology) were consulted to determine their experience with adhesive sealing packages and to discuss the sealing procedures and testing methods being used. Information was obtained from informal conversations with key engineers at the selected companies. This had the advantage over a formal questionnaire in that the information obtained was generally candid, but the disadvantage that the same type of information was not obtained from the various individuals contacted and consequently presented a collation problem. The companies contacted were:

Analog Devices Corporation, Wilmington, Massachusetts  
Beckman Instruments, Inc., Helipot Division, Fullerton, California  
Burr-Brown, Tucson, Arizona  
Collins Radio Group, Rockwell International, Dallas, Texas  
General Dynamics, Pomona, California  
Hewlett-Packard, Colorado Springs, Colorado  
Hewlett-Packard, Loveland, Colorado  
Hewlett-Packard, Palo Alto and Santa Rosa, California  
IBM, Huntsville, Alabama and Owego, New York  
Jet Propulsion Laboratory, Pasadena, California  
Microwave Semiconductor Corporation, Somerset, New Jersey  
Texas Instruments, Dallas, Texas

Results are summarized in Table 1. Numbering in this table does not correspond to the listing given above. A summary of conclusions drawn from this survey are as follows:

- ° Hybrid manufacturers want to accept adhesive sealing because of its obvious advantages over metallurgical sealing (e.g., lower cost, simpler processing, less chance of introducing contaminants, easier rework).
- ° Most hybrid manufacturers believe that adhesive sealed packages are capable of or, with improvements, will be capable of meeting MIL-STD-883A requirements. In many cases, these companies have performed tests and accumulated field performance data substantiating this.
- ° Even hybrid manufacturers who believe that adhesive sealed packages cannot be hermetic in the sense that metallurgically sealed packages are, believe that they are acceptable for many applications where moisture environments are not severe.

Of those individuals contacted, all were either optimistic or enthusiastic. Some felt that adhesive sealing would be proven adequate for many applications while others felt that adhesive sealing already was capable of meeting the MIL-STD-883A requirements and acceptable as an alternate to metallurgical sealing.

The survey also indicated that hybrid manufacturers were divided on the superiority of paste or film adhesives for package sealing. The most widely

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TABLE I. SUMMARY OF RESULTS OF SURVEY OF HYBRID MICROCIRCUIT MANUFACTURERS

COMPANY	ADHESIVE SEALING EXPERIENCE	PACKAGE SIZE	SEAL WIDTH	SEAL TYPE	ADHESIVES EVALUATED	ADHESIVE SELECTED	REASON
1	Several Years	2.54 cm (1 inch) square	0.102 cm (40 mils)	Ceramic/ceramic, gold plated Kovar/gold plated Kovar	Ablefilm 529 Ablefilm 550	Ablefilm 529	Ablefilm 529 better to seal has good interhesion but grades rapidly during humidity cycling.
2	No Previous	0.953 by 1.59 cm (3/8 by 5/8 in)	0.076 cm (30 mils)	Ceramic/ceramic. Package consists of a ceramic substrate with circuitry on it, a ceramic frame, and a ceramic lid. Thus it has two adhesive seals.	Ablefilm 517A Ablefilm 529 Ablefilm 532 Ablefilm 535 (ECF) Ablefilm 550	Ablefilm 550	
3	8 Years	3.30 by 3.81 cm (1.3 by 1.5 in)		Ceramic/Kovar		Eccobond 104	
4	Some	Two Sizes 1.59 cm (0.625 in) square 1.27 by 2.54 cm (0.5 by 1 inch)	0.152 cm (60 mils) 0.102 cm (40 mils)	Ceramic/ceramic	Epo-Tek H72 Ablefilm 529 Ablefilm 532	Ablefilm 513, which is a modification of Ablefilm 532 containing less filler and no thickening agent.	Epo-Tek H72 difficult to work with since it is a paste. Ablefilm 529 was rejected because it contains acetone.

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## IT MANUFACTURERS CONCERNING ADHESIVE PACKAGE SEALING

REASON	PREFORM THICKNESS	PACKAGE ASSEMBLY/SEAL PROCEDURE	PRESSURE DURING CURE (SEAL AREA)	APPLICATION	COMMENTS
efilm 550 adheres ter to gold and good initial ad- des rapidly ing humidity ling.	0.018 cm (7 mils)	A special alignment fixture is used to assemble the packages. The preforms are placed on the substrates and then the lids are positioned. The packages are cured in nitrogen at 120°C. They do not use a breather hole, and consequently, no vacuum bake-out.	1 to 1.5 psi or 0.69 to $1.03 \times 10^5 \text{ N/m}^2$	Military	Tests for moisture permeation were made by sealing transistor devices with un-passivated aluminum metallization in packages and subjecting them to 30-40 cycles of humidity per MIL-STD-883A. No changes in the leakage current of the reversed biased transistors or corrosion of the aluminum metallization was observed.  Recommend preforms be at least 0.120 cm (40 mils) wide.
	0.013 cm (5 mils)	Purge and backfill with nitrogen through adhesive.	1 lb/total seal area, so 16.7 psi or $1.15 \times 10^5 \text{ N/m}^2$ .	Military	Package flatness is important and should be no worse than 0.003 cm/cm (3 mils/inch).  After sealing, packages contain about 4 micrograms of water.
	Not applicable		Zero	Commercial	Metallization is thin film gold, and the hybrid contains tantalum nitride resistors, custom MOS, operational amplifiers, and diodes. Interconnections are ultrasonically bonded 0.004 cm (1.5 mil) aluminum wires.  Experience with this package has been excellent.
Tek H72 was icult to work since it is a t. Ablefilm as rejected se it outgasses me.	0.020 to 0.023 cm (8 to 9 mils)	Packages are assembled in room environment, clamped with binder clips, and cured in a nitrogen oven. They do not purge or back-fill the packages with nitrogen.	1 lb/linear inch seal, so 16.7 to 25 psi or 1.15 to $1.72 \times 10^5 \text{ N/m}^2$ .	Commercial	Ablefilm 513 presently is being evaluated for sealing gold plated Kovar lids to ceramic substrates.  Since there is an alignment problem (accuracy is 0.038 cm or 15 mils) and since space is not critical, the seal width of the second package is being increased to 0.152 cm (60 mils) also.  Unable to measure fine leak rates reliably because of helium adsorbed or absorbed by adhesive, so test for gross leaks only.

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TABLE I. SUMMARY OF RESULTS OF SURVEY OF HYBRID MICROCIRCUIT MANUFACTURERS

COMPANY	ADHESIVE SEALING EXPERIENCE	PACKAGE SIZE	SEAL WIDTH	SEAL TYPE	ADHESIVES EVALUATED	ADHESIVE SELECTED	REASON
5	3 Years	Perimeters ranging from 6.4 to 7.6 cm (2.5 to 3 inches) up to about 17.8 cm (7 inches)		Gold plated Al/Gold plated Al		Ablefilm 535 (ECF)	
6	Has used them in the past.						
7	6 Years	2.29 cm (0.9 in) square	0.127 cm (50 mils)	Ceramic/ceramic. Seal is made over 0.018 cm (7 mils) thick leads.	Nine epoxy adhesives	Ablefilm 507	

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## CIRCUIT MANUFACTURERS CONCERNING ADHESIVE PACKAGE SEALING (CON'T)

REASON	PREFORM THICKNESS	PACKAGE ASSEMBLY/SEAL PROCEDURE	PRESSURE DURING CURE (SEAL AREA)	APPLICATION	COMMENTS
Unknown				Military (Microwave Hybrids)	<p>No reports of field failures, so apparently package is reliable.</p> <p>Typical measured helium leak rates for the larger packages are in the <math>10^{-8}</math> to <math>5 \times 10^{-7}</math> atm cc/sec range.</p> <p>Package flatness must be no worse than about 0.002 cm/cm (2 mils/inch).</p> <p>Sealing yield is at least 90%. Packages that do not pass initial leak test are touched up with Ablebond 660-3 (electrically insulative) or Ablebond 36-2 (electrically conductive).</p> <p>If packages must be reworked, they are heated to 125-150°C, opened, and the adhesive peeled off. This procedure permits the packages to be reused.</p> <p>Moisture content is determined by monitoring diode leakage current to determine dew point (MIL-STD-883A, Method 1013). Typically dew points are around -70°C indicating a moisture content of about 2 ppm.</p>
0.038 cm (15 mils)	Packages are assembled by hand, flat, rigid spacers (or force spreaders) are placed on each side and these assemblies are held together by spring clamps. The packages are assembled in room ambient and cured in a nitrogen environment. Prior to reaching the cure temperature, the packages are purged and back-filled with nitrogen through the adhesive seal.	10 psi or $6.9 \times 10^4$ N/m <sup>2</sup>	Commercial	<p>No plans to use adhesives for sealing military hybrids. Think it will be several years before adhesive sealing is accepted by the various agencies.</p> <p>Have made moisture content analyses on both metallurgically sealed and adhesive sealed packages. Typical values for freshly sealed specimens were around 100 ppm for metallurgically sealed packages and from 200 to 400 ppm for adhesive sealed packages. Feel the additional moisture is coming from the adhesive and probably could be reduced by proper process controls.</p> <p>Feel adhesive sealed packages cannot be hermetic to extent that metallurgically sealed packages are, but also feel that they will be adequately hermetic for many applications and that economic considerations will lead to their acceptance in these cases.</p>	

TABLE I. SUMMARY OF RESULTS OF SURVEY OF HYBRID MICROCIRCUIT MANUFACTURERS

COMPANY	ADHESIVE SEALING EXPERIENCE	PACKAGE SIZE	SEAL WIDTH	SEAL TYPE	ADHESIVES EVALUATED	ADHESIVE SELECTED	REASON
8	No previous	3.5 cm (1-3/8 in) square	0.19 cm (75 mils)	Gold Plated Kovar/Gold Plated Kovar	Ablefilm 529 Ablefilm 550	Ablefilm 550	Ablefilm 529 was rejected because it flakes and is more difficult to remove.
9	1 Year	Two Sizes 0.635 cm (1/4 in) diameter 0.953 cm (3/8 in) diameter	0.064 cm (25 mils)	Ceramic/ceramic. Seal is made over 0.015 cm (6 mils) thick leads. Total seal thickness is 0.020 to 0.023 cm (8 to 9 mils)	Ablefilm 535 Ablefilm 542 Epo-Tek H77	Epo-Tek H77	Film adhesive seals were seriously degraded by exposure to 85°C/85% RH environment and by temperature cycling between -65°C and +200°C.
10	Several years	Various	0.102 cm (40 mils)	Plastic/plastic Ceramic/Ceramic	Film epoxies Paste epoxies	Epo-Tek H77	Epo-Tek H77 has good moisture resistance and does not cause corrosion. Film adhesives are relatively expensive, must be kept frozen, have a relatively short shelf life, and are easily damaged. Some also contain undesirable solvents.

## NUFACTURERS CONCERNING ADHESIVE PACKAGE SEALING (CON'T)

SON	PREFORM THICKNESS	PACKAGE ASSEMBLY/SEAL PROCEDURE	PRESSURE DURING CURE (SEAL AREA)	APPLICATION	COMMENTS
529 was re- cause it d is more to rework.	0.020 cm (8 mils)	The packages are assembled and cured in a jiggling fixture which serves both to align the parts and to apply a force during cure. The lids are placed in the jiggling fixture and the preforms are aligned on top on them and put in a vacuum at 120°C for 10 minutes to remove the residual solvents from the adhesive. Then package assembly is completed and the packages are cured in a nitrogen environment for 2 hours at 150°C.	900 grams/total seal area. So 4.85 psi or $3.3 \times 10^4$ N/m <sup>2</sup> .	Military	Engineering development completed. Moving into the pilot line phase.  The package is a Bendix package designed for solder sealing. Appears very rugged and is made of ceramic with a gold plated Kovar seal rim. Lid also is gold plated Kovar.  Important to control cure schedule carefully. Ablefilm 550 will not cure (in two hours) at temperatures up to 135°C, and the cured adhesive loses its bond strength at around 160°C.  Package and lid flatness is important in obtaining good seals. Packages are flat to about 0.0025 cm (1 mil).  Motivation for going to adhesive sealing is the necessity to rework expensive packages and to reduce particulate contaminants within the packages.
isive seals ously de- exposure 5% RH en- and by re cycling 65°C and	Not applicable.	They apply the adhesive to the lids by doctor-blading a thin layer on a smooth surface and dipping the lids into it. Then they assemble the packages in a nitrogen environment and wait about 20 minutes to allow the adhesive to flow around the leads before curing.	Zero		Satisfied with Epo-Tek H77 but currently evaluating another paste adhesive made by Ablestik (probably Ablebond 789).  Packages sealed with Epo-Tek H77 cannot be reworked except for touch-up of leakers.  Typical leak rates are $5 \times 10^{-7}$ atm cc/sec or less.  Found must wait 5 or 10 minutes before fine leak testing to eliminate the effects of adsorbed and absorbed helium.  Presently are aging packages under room ambient and remeasuring leak rates to determine retention of seal integrity. No evidence of seal degradation after 1500 hours.
177 has good resistance not cause . Film ad- ire rela- pensive, ept frozen, atively lf life, usily Some also ndesirable	Not applicable.	The adhesive is applied to the lids by off-set printing and the packages are placed on the lids and cured in an inverted position to keep the adhesive from flowing into the circuitry. Since the adhesive is a paste, pulling a vacuum of any kind would suck out the adhesive, so the packages cannot be purged and back-filled through the uncured adhesive as is often done when film adhesives are used.	100 grams/linear inch. So 5.5 psi or $3.8 \times 10^4$ N/m <sup>2</sup> .	Commercial	Bad flow-out problem with Epo-Tek H77 and special precautions must be taken to control it and/or to avoid deleterious effects due to it.  Packages sealed with Epo-Tek H77 cannot be reworked.  Packages have a small breather hole in the bottom. After cure and a 24 hour, 150°C bake-out, this hole is sealed with Eccoseal 285 (Catalyst #9).  Packages sealed in this manner meet or exceed the seal requirements of MIL-STD-883A and retain their integrity after 10 temperature cycles between -65°C and +150°C and after being subjected to the MIL-STD-883A ten day humidity test. Leak rates are measured using Krypton-85.

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TABLE I. SUMMARY OF RESULTS OF SURVEY OF HYBRID MICROCIRCUIT MANU

COMPANY	ADHESIVE SEALING EXPERIENCE	PACKAGE SIZE	SEAL WIDTH	SEAL TYPE	ADHESIVES EVALUATED	ADHESIVE SELECTED	REASO
11	6 or 7 Years	Small 3.5 cm (1-3/8 in) square	0.19 cm (75 mils)	Ceramic/Ceramic (Integral Lead Type) Gold Plated Kovar/Gold Plated Kovar	Ablefilm 550	Ablefilm 550	Packages seal the seal requirements STD-883A, their integrity temperature between -55 +125°C, and MIL-STD-883 humidity test
12	Over a year	Range from 1.27 by 1.91 cms (1/2 by 3/4 in) to 3.81 by 5.72 cms (1-1/2 by 2-1/4 in)	0.15 to 0.20 cms (60 to 80 mils)	Ceramic/Ceramic Seal is made over thick film gold conductors		Ablefilm 529	

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## CIRCUIT MANUFACTURERS CONCERNING ADHESIVE PACKAGE SEALING (CON'T)

REASON	PREFORM THICKNESS	PACKAGE ASSEMBLY/SEAL PROCEDURE	PRESSURE DURING CURE (SEAL AREA)	APPLICATION	COMMENTS
Packages sealed with Ablefilm 550 meet the seal test requirements of MIL-STD-883A, retain their integrity when temperature cycled between -55°C and +125°C, and pass the MIL-STD-883A ten day humidity test.	Not applicable	Packages are assembled in room ambient.	Zero	Commercial/Military (Ground Test Equipment)	Field experience has been excellent.
	0.020 cm (8 mils)			Military	<p>Packages become leakers when subjected to a 150°C stabilization bake. Probably due to fact that the lids are somewhat warped, and the resulting stresses relax when the adhesive softens.</p> <p>Also, packages fail when subjected to an 85°C/85% RH environment, but will survive long term exposure to a 50°C/85% RH environment.</p> <p>Moisture permeates the seal. Feel this is an inherent characteristic of all organics. Feel that given sufficient time, the moisture partial pressure inside the packages will equal that of the surrounding environment and that the most that can be expected of any adhesive seal is that it will serve a dampening function to moderate the moisture content in the packages since permeation is a rather slow process.</p> <p>Also feel that at present inadequate information exists to say what moisture content degrades the performance of particular hybrid microcircuits, and that until this situation is resolved and an accurate reproducible method of measuring moisture content is developed, it is impossible to accurately judge the applications for which adhesive sealing is appropriate.</p>
	0.015 cm (6 mils)			Commercial	<p>No failure reports to date.</p> <p>Feel adhesive sealing is adequate for real world conditions. However, also feel that no adhesive will give a truly hermetic seal and so have a company policy not to use adhesives to seal packages for military applications.</p> <p>Gross leak test all packages and periodically sample fine leak test. Experience shows adhesive sealing does not result in fine leakers. That is, if the packages leak at all, they will be gross leakers, so fine leak testing is unnecessary.</p> <p>Performed tests using packages containing transistors and thin film nichrome resistor networks, and looked for effects on <math>I_{CBO}</math> and resistance, respectively. The requirement for the resistor network is stability to 0.01%. Testing included the ten-day humidity test, a 168 hour/125°C life test, and temperature cycling between -65°C and +150°C. Packages retained their seal integrity (i.e., passed the MIL-STD-883A seal test) after all tests. Only test causing deleterious effect was the ten-day humidity test. Excessive moisture entered the package during this test as indicated by higher <math>I_{CBO}</math> values, but did not effect the thin film nichrome resistor network. Packages also have been subjected to long term exposure to 98% relative humidity at room temperature with no adverse effects.</p> <p>General opinion is that while adhesive sealed packages are not hermetic in the sense that moisture is excluded from them and are not capable of withstanding all of the test environments of MIL-STD-883A, they are adequate for use under the real world conditions to which hybrids are exposed.</p>

used adhesives are the films, Ablefilm 529 and 550, and the paste, Epo-Tek H77. Hybrid manufacturers who have tested both Ablefilm 529 and 550 also were divided as to which was better. The following were general points of agreement:

- ° Pressure must be applied to the film adhesives during cure.
- ° The seal should be as wide as possible.
- ° Care must be exercised in package assembly.
- ° Package (and lid) flatness is a critical parameter which can make the difference between success or failure.

In general, each hybrid manufacturer has developed his own specific package assembly and seal procedure details: e.g., fixturing, assembly and sealing ambient (air or nitrogen environment), sealing pressure, seal width, vacuum bake-out, purging and back-filling, and cure schedule.

A significant conclusion reached from the survey was that the use of adhesives for package sealing is promising and that further evaluation for possible qualification of adhesive sealing as an alternate to metallurgical sealing should be conducted. This evaluation should include a thorough investigation of the effects of various parameters on the integrity and quality of adhesive seals, and establishing minimum acceptable values for them.

## 2.2 COST COMPARISON OF METALLURGICAL VERSUS ADHESIVE PACKAGE SEALING

Cost comparisons of metallurgical and adhesive package sealing methods for three different situations were made. Results are presented in Tables 2 through 4. While comparisons such as these, based largely on material and labor costs, are valuable, they are incomplete since they do not take into consideration less tangible cost factors such as the impact on reliability, design flexibility, or reworkability.

In Table 2, a comparison is given between seam sealing and adhesive sealing for a 2.54 cm (1 inch) square, 32-lead, gold-plated Kovar flatpack-type package. Assuming that processing costs (cleaning and sealing) and yields are the same for the two methods, the explicit savings associated with adhesive sealing is the lower material cost of the lid and seal preform.

Table 2. Cost Comparison\*

Metallurgical Versus Adhesive Package Sealing  
 32 Lead Flatpack-Type Package 2.54 cm (1 inch) Square  
 (Quantities of 250 to 500 Packages)

Material	Seam Sealed	Adhesive Sealed
Package	\$ 9.92	\$ 9.92
Lid		
Gold Plated Kovar	1.74	0.16
Ceramic		
Preform		
Gold/Tin	1.48	0.23
Adhesive		
TOTAL	\$13.14	\$10.31

\* Processing costs are not included; processing costs and yields are assumed to be the same for both methods.

Table 3. Cost Comparison\*

Metallurgical Versus Adhesive Package Sealing  
 32 Lead Integral Lead-Type Package 2.54 cm (1 inch) Square  
 (Quantities of 250 to 500 Packages)

Material/Labor	Metallurgically Sealed	Adhesive Sealed
Lid		
Gold Plated Kovar	\$ 2.13	\$ 0.36
Ceramic		
Preform		
Gold/Tin Adhesive	1.48	0.23
Lead Frames	2.18	2.18
Dielectric & Seal Ring**	0.60	NR
Lead Frame Bonding**	0.98	0.98
TOTAL	\$ 7.37	\$ 3.75

\*Assumes sealing labor costs and yields are the same for the two methods.

\*\*Assumes a mature process capability so that standard hours times labor rate can be used.

Table 4. Cost Comparison\*

**Metallurgically Sealed Flatpack-Type Package  
Versus  
Adhesive Sealed Integral Lead-Type Package  
(Quantities of 250 to 500 Packages)**

Material/Labor	Flatpack-Type	Integral Lead-Type
Package	\$ 9.92	NR
Lid		
Gold Plated Kovar Ceramic	1.74	\$ 0.36
Preform		
Gold/Tin Adhesive	1.48	0.23
Lead Frames	NR	2.18
Substrate Bonding	0.77**	NR
Wire Bonding to Terminals	10.24**	NR
Lead Frame Bonding	NR	0.98***
<b>TOTAL</b>	<b>\$24.15</b>	<b>\$ 3.75</b>

\*Assumes sealing labor costs and yields are the same for the two methods.

\*\*Assumes a mature process capability so that standard hours times labor rate can be used. The cost of wire bonding could be considerably less depending on local labor rates and whether the work is done on-shore or off-shore.

\*\*\*Lead frame bonding assumes automatic gang bonding whereas wire bonding is performed point-by-point.

In seam sealing, the cover must be metal (e.g., gold-plated Kovar). If adhesive sealing is used, the cover can be ceramic. Furthermore, a gold/tin preform is required for seam sealing whereas an adhesive preform is required for adhesive sealing. The difference in cost of these two items (\$2.83), while not insignificant, may be considered small when compared to the total cost of the hybrid. The cost difference is further reduced to \$1.35 if the package is welded (eliminating the necessity for the gold/tin preform).

However, there is the important consideration of package reworkability. If the package is brazed and rework is required, the package may possibly be salvaged but certainly not the lid or preform (\$3.22). If the package is welded and rework is required, then both the package and lid must be replaced (\$11.66), the substrate must be rebonded to the package (\$0.77) and interconnections must be made from the substrate to the package terminals (\$10.24), for a total of \$22.67. In addition, unless great care is exercised, there is a high probability that the substrate will be damaged when it is removed from the package. Consequently, if rework is desired and seam sealing is used, brazing is the only practical method, which is why this method was the one priced in Table 2. In this case, rework after sealing would cost \$3.22. On the other hand, if the package is adhesive-sealed, rework would cost \$0.39 - the cost of a new ceramic lid and an adhesive preform.

Table 3 shows a comparison of metallurgical and adhesive sealing for a 2.54 cm (1 inch) square, 32-lead, integral-lead type package. In both cases, such a package is lower in cost than the gold-plated Kovar flatpack-type package. As shown, the explicit savings in adhesive sealing is \$3.62 if it is assumed that the metallurgical seal is made using a gold/tin preform.

There are other important advantages resulting from adhesive sealing. Adhesive-sealed integral lead packages can be used for either thin or thick film circuitry while metallurgical sealing can be used only for thick film integral lead circuits since a dielectric and seal ring are required which must be fired at a high temperature. Thus adhesive sealing permits greater flexibility in circuit design. Also, if the metallurgical seal is made with a gold/tin preform, the package must be passed through a belt furnace and

subjected to temperatures in excess of 300°C. This restricts both the types of electronic components which can be used and the methods for mounting them. Lower sealing temperatures can be achieved by using a solder preform instead of one of gold/tin, but this introduces potential problems of contaminating the circuit with flux and/or introducing solder balls. In any case, adhesive-sealed packages can be easily reworked at a material cost of \$0.59, while rework of the metallurgically sealed package, if it is possible, requires a material cost ranging from \$2.13 to \$3.61.

Table 4 shows a comparison of the two cost extremes - a seam-sealed, gold-plated Kovar flatpack and an adhesive-sealed, integral lead-type package. The explicit savings resulting from using the adhesive sealed integral lead-type package is substantial - \$20.40. As discussed in conjunction with Table 2, for the metallurgically sealed, gold-plated Kovar flatpack, rework costs range from \$3.22 if the seal is made by brazing to \$22.67 if the seal is made by welding. On the other hand, rework of the adhesive sealed integral lead package would cost \$0.59 - the cost of a new ceramic lid and an adhesive preform.

## 2.3 EVALUATION OF ADHESIVES FOR PACKAGE SEALING

### 2.3.1 Preliminary Laboratory Studies

Prior to beginning the formal laboratory evaluation of adhesive sealing, a preliminary study was made using available in-house packages. The objective of this effort was to gain experience in adhesive sealing, to determine how difficult it is to obtain seals which meet MIL-STD-883A seal requirements, and to obtain insight concerning the long-term retention of integrity that can be expected of adhesive seals.

The package selected for the initial attempt at sealing is shown in Figure 1. This package consisted of a small integral lead alumina substrate, 0.81 cm (320 mils) wide and 2.45 cm (965 mils) long, and a solder-plated Kovar lid. The substrate had a 0.076 cm (30 mils) wide thick film platinum/gold seal ring to accommodate the lid. The lid was 0.76 cm (300 mils) wide by 1.98 cm (780 mils) long (outside dimensions) and had a 0.089 cm (35 mils)

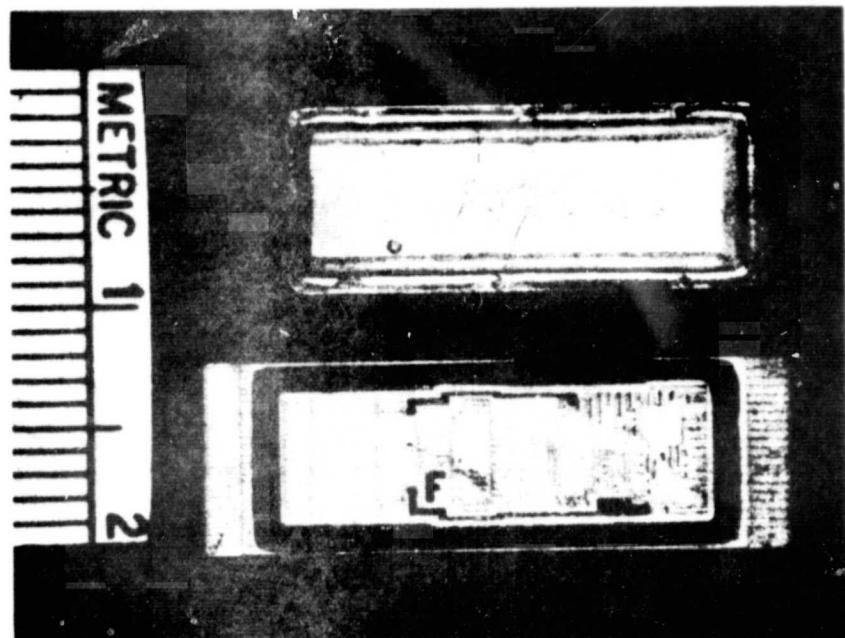


Figure 1. Small Integral Lead Package

wide lip. This package was designed for solder sealing using the GTI sealer.

Ten packages were sealed using 0.013 cm (5 mils) thick Ablefilm 529 preforms which had been hand cut and were 0.076 to 0.089 cm (30 to 35 mils) wide. The packages were clamped and the adhesive cured for two hours at 150°C in nitrogen. Measured leak rates, after bombing the packages in helium at 30 psig for three hours, are shown in Table 5. The allowed maximum leak rate for a package of this size (internal volume of 0.115 cc) as calculated from the equation given in Method 1014.1 of MIL-STD-883A, is  $6.8 \times 10^{-8}$  atm cc/sec (air equivalent). The measured leak rates for all packages were within this requirement.

To determine the effect of seal width on seal quality, an additional eight packages were sealed using preforms only 0.051 cm (20 mils) wide instead of 0.076 to 0.089 cm (30 to 35 mils) wide. Measured leak rates for these packages are given in Table 6. Only two of these packages had leak rates which met the MIL-STD-883A seal requirements. All others had excessive leak rates. The important conclusion from this investigation is that seals meeting the seal requirements of MIL-STD-883A can be reliably obtained using an epoxy adhesive, but that care must be exercised to assure the selection of proper seal parameters such as seal width.

Another set of larger integral lead packages, 2.29 cm (0.9 inch) square, sealed with Ablefilm 507 also were leak tested after being bombed in helium at 30 psig for three hours. These packages were all ceramic (i.e., both substrates and lids were ceramic). Results obtained are shown in Table 7. The maximum permitted leak rate for these packages (internal volume of 1.05 cc) was calculated to be  $7.5 \times 10^{-7}$  atm cc/sec (air equivalent). The measured leak rates of all packages are well within this value.

In addition, the ambient gases of two of the packages (packages 1 and 2) were analyzed by gas chromatography (GC) to determine their moisture content. Results showed no measurable moisture within the sensitivity of the test

Table 5. Seal Test Results for Small Integral Lead Packages Sealed With 0.076 to 0.089 cm (30 to 35 mils) Wide Ablefilm 529 Preforms

Package	Measured Leak Rate Air Equivalent (atm cc/sec)
1	$2 \times 10^{-9}$
2	$<0.2 \times 10^{-9}$
3	$1.5 \times 10^{-8}$
4	$1.2 \times 10^{-8}$
5	$6 \times 10^{-9}$
6	$7.2 \times 10^{-9}$
7	$2 \times 10^{-9}$
8	$0.2 \times 10^{-9}$
9	$1.4 \times 10^{-8}$
10	$5 \times 10^{-9}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $6.8 \times 10^{-8}$  atm cc/sec (air equivalent).

Table 6. Seal Test Results for Small Integral Lead Packages Sealed With 0.051 cm (20 mils) Wide Ablefilm 529 Preforms

Package	Measured Leak Rate Air Equivalent (atm cc/sec)
1	$1 \times 10^{-7}$
2	$1.3 \times 10^{-7}$
3	$6.4 \times 10^{-8}$
4	$1.3 \times 10^{-7}$
5	$1.2 \times 10^{-7}$
6	$7 \times 10^{-9}$
7	$1.4 \times 10^{-7}$
8	$1 \times 10^{-7}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $6.8 \times 10^{-8}$  atm cc/sec (air equivalent).

Table 7. Seal Test Results for Newly Prepared 2.29 cm (0.9 inch) Square Integral Lead Ceramic Packages

Package	Measured Leak Rate Air Equivalent (atm cc/sec)
1	$1.2 \times 10^{-8}$
2	$1.8 \times 10^{-8}$
3	$1.2 \times 10^{-8}$
4	$1.1 \times 10^{-8}$
5	$1.8 \times 10^{-8}$
6	$1.6 \times 10^{-8}$
7	$1.6 \times 10^{-8}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $7.5 \times 10^{-7}$  atm cc/sec (air equivalent).

method (i.e., less than 10 ppm). The elapsed time between package sealing and testing was six weeks. Thus, the results not only show that seals meet MIL-STD-883A seal requirements, but also that they have a low moisture permeation rate.

The seals for these packages were formed over 0.018 cm (7 mils) thick lead frames using 0.038 cm (15 mils) thick adhesive preforms. Typical leak rates for newly-sealed packages after helium bombing at 30 psig for three hours were 1 to  $2 \times 10^{-8}$  atm cc/sec (air equivalent). To check long-term retention of seal integrity, eight "old" packages of this type were obtained and their leak rates measured. The history of these packages, except for their date of manufacture, is not known. Some may have been stored in a factory environment while others may have been removed from field hardware. The year of manufacture for these packages and their present measured leak rates after helium bombing for three hours at 30 psig are given in Table 8. The leak rates for all of these packages were found to be within the allowed rate of  $7.5 \times 10^{-7}$  atm cc/sec (air equivalent). Considering the handling and mishandling which these packages probably received and the age of some of the packages (six years or more), this is a relatively impressive testimony to the long-term retention of seal integrity of epoxy-sealed packages.

These low leak rates as determined from helium measurements do not necessarily mean that moisture has not entered the packages. As discussed in the Appendix, the rates of helium permeation and moisture permeation are not directly correlatable. However, several packages were opened and visually inspected for evidence of corrosion due to the presence of moisture. The circuits contained four IC chips, two chip capacitors, and thin film aluminum metallization with gold wire interconnections. No evidence of corrosion was found, an indication that the packages had not contained excessive moisture.

### 2.3.2 Formal Laboratory Evaluation

The objective of this effort was to determine if adhesive sealed packages can pass the seal test requirements of MIL-STD-883A, Method 1014.1 after

Table 8. Seal Test Results for Old  
2.29 cm (0.9 inch) Square  
Integral Lead Ceramic Packages

Year of Manufacture	Measured Leak Rate Air Equivalent (atm cc/sec)
Prior to 1969	$4.4 \times 10^{-7}$
1969	$3.0 \times 10^{-8}$
1969	$1.7 \times 10^{-7}$
1969	$2.6 \times 10^{-7}$
1972	$4.2 \times 10^{-7}$
1974	$3.4 \times 10^{-8}$
1974	$2.1 \times 10^{-7}$
1974	$4.2 \times 10^{-8}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $7.5 \times 10^{-7}$   
atm cc/sec (air equivalent).

they have been subjected to selected MIL-STD-883A screen tests. Parameters evaluated were package size, adhesive material, and (in the case of film adhesives) pressure applied during cure and preform thickness.

Gold-plated Kovar flatpacks with solid ring frames were selected for this evaluation because of their wide use in NASA/MSFC equipment. Three film adhesives (Ablefilm 507, 529 and 550) and one paste adhesive (Epo-Tek H77) were selected for evaluation on the basis of the survey of adhesive and hybrid manufacturers. Tests were made to evaluate and compare the performance of these adhesives, and to determine the effects of preform thickness and clamping pressure during cure on the seal quality of the film adhesives. Seal integrity was determined by performing fine and gross leak tests in accordance with MIL-STD-883A, Method 1014.1, Test Conditions A<sub>2</sub> and C<sub>1</sub>, respectively. For the fine leak test the packages were bombed at 30 psig helium for 3 hours.

Testing consisted of sequentially subjecting the packages to the following MIL-STD-883A Class A screen tests.

- Thermal Shock, Method 1011.1, Test Condition A (i.e., 15 cycles between 0°C and +100°C)
- Temperature Cycling, Method 1010.1, Test Condition C, except 15 cycles (i.e., 15 cycles between -65°C and +150°C)
- Mechanical Shock, Method 2002.1, Test Condition B (i.e., 5 shock pulses at 1500 g's in the Y<sub>1</sub> plane)
- Constant Acceleration, Method 2001.1, Test Condition B (i.e., 10,000 g's in the Y<sub>1</sub> plane)

Seal integrity was determined initially and after subjecting the packages to each of the above test environments.

#### 2.3.2.1 Package Cleaning and Assembly Methods

The packages (and lids) were cleaned by brushing in deionized water, acetone, and isopropyl alcohol and spraying with Freon TF. Cleaned packages

were then stored in a chamber containing a nitrogen ambient, and again sprayed with Freon TF immediately prior to use. Packages were assembled within a few hours after they were cleaned. The adhesive preforms were removed from the freezer and placed in the nitrogen chamber with the packages and allowed to stand at room temperature for approximately one hour before they were used. The packages were assembled by hand in room ambient.

In the case of the film adhesives, the preforms were carefully aligned on the lids and placed on a hot plate at approximately 120°C until they softened and adhered to the lids, a procedure which took only a few minutes. The lids were allowed to cool and then the packages were carefully aligned on top of them. Teflon coated stainless steel plates, 0.102 cm (40 mils) thick and the same size as the packages, were placed on the tops and bottoms of the assembled packages. Spring clamps were then placed on these assemblies to hold them together. These units were carefully placed on a tray and put into a nitrogen oven to cure. The Teflon coated plates served as force spreaders to assure that the packages (and lids) remained flat during cure. This precaution was taken to avoid "oil-canning" of the package which could cause a bad seal or set up stresses which might be relieved later and cause loss of seal integrity.

In the case of the paste adhesive, the adhesive was applied to the package rims by touching the packages against a glass slide on which a 0.009 cm (3.5 mils) thick layer of adhesive had been doctor-bladed. The packages were then carefully positioned on the lids and assembly was completed following the same procedure as used for the packages sealed with film adhesives. In this case since the viscosity of the adhesive is initially very low, the clamps used to hold the packages together provided only sufficient force to assure that the parts remained in alignment during cure.

### 2.3.2.2 Packages Evaluated

Originally it was planned to use 1.27 cm (1/2 inch) square and 5.08 cm (2 inch) square gold-plated Kovar packages. Package blanks and lids of these sizes were ordered from Isotronics. Package blanks are identical to

completed packages except that the holes for the feedthroughs have not been drilled and the feedthroughs installed. They were less expensive than the completed packages, and were suitable for the investigation since the evaluation of package sealing required no electrical measurements. The use of package blanks is preferable to the use of packages since there are no feedthroughs to be possible sources of leaks. Any leak rate measured must be due to the package seal.

Because of the long delivery time of the packages ordered from Isotronics (>15 weeks), NASA supplied 2.54 cm (1 inch) square gold-plated Kovar packages for evaluation. While some work was done using the 5.08 cm (2 inch) square packages, the major effort was made using the 2.54 cm (1 inch) square and the 1.27 cm (1/2 inch) square packages.

### 2.3.2.3 Effect of Sealing Pressure

The first set of tests performed was an investigation of the effect of pressure applied during cure on the quality of seals obtained when film adhesives were used to seal packages. This data was desired to select an appropriate sealing pressure to be used in further tests comparing the film adhesives with each other and with the paste adhesive.

Twelve of the 2.54 cm (1 inch) square packages were sealed using 0.015 cm (6 mils) thick Ablefilm 507 preforms. Three each were cured with pressures of 3.5, 6.9, 10.3 and  $13.8 \times 10^4 \text{ N/m}^2$  (5, 10, 15 and 20 psi) applied to the seal areas. All packages were assembled in room ambient and cured for two hours at 165°C in nitrogen. Seal testing after bombing at 30 psig helium for three hours gave the results shown in Table 9. Measured leak rates are given as air equivalents. The permitted maximum measured leak rate for these packages (internal volume of 1.39 cc) was calculated from the equation given in Method 1014.1 of MIL-STD-883A to be  $5.7 \times 10^{-7} \text{ atm cc/sec}$  (air equivalent). Good seals were obtained at all four of the selected seal pressures. The results obtained for the packages sealed at 3.5, 6.9, and  $10.3 \times 10^4 \text{ N/m}^2$  (5, 10 and 15 psi) are essentially identical, but the leak rates obtained for those sealed at  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) are a little higher.

**Table 9. Seal Test Results (Initially and After Thermal Shocking) for 2.54 cm (1 inch) Square Gold Plated Kovar Packages Sealed With 0.015 cm (6 mils) Thick Ablefilm 507 Preforms With Four Different Pressures Applied to the Seal Area**

Sealing Pressure	Measured Leak Rate Air Equivalent (atm cc/sec)
$3.5 \times 10^4 \text{ N/m}^2$ (5 psi)	$4.2 \times 10^{-8}$ $4.6 \times 10^{-8}$ $8.4 \times 10^{-8}$
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$4.0 \times 10^{-8}$ $4.2 \times 10^{-8}$ $5.8 \times 10^{-8}$
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$3.8 \times 10^{-8}$ $4.8 \times 10^{-8}$ $10.0 \times 10^{-8}$
$13.8 \times 10^4 \text{ N/m}^2$ (20 psi)	$5.6 \times 10^{-8}$ $8.8 \times 10^{-8}$ $9.8 \times 10^{-8}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $5.7 \times 10^{-7}$   
atm cc/sec (air equivalent).

These packages were then thermal shocked for 15 cycles between 0°C and +100°C by immersing them in ice water and boiling water. Subsequent seal testing showed that all but one were gross leakers. The only package that survived was the first  $6.9 \times 10^4 \text{ N/m}^2$  (10 psi) specimen listed in Table 9. Its measured leak rate remained the same ( $4.0 \times 10^{-8} \text{ atm cc/sec}$ ). This result was not expected since shocking between 0°C and +100°C is not considered to be a particularly severe stress environment. A possible explanation is that when the packages were immersed in the boiling water, water was absorbed causing either hydrolytic degradation or swelling of the epoxy adhesive. Consequently, new packages were prepared and thermal shocked using fluorocarbons, FC40 and FC77, as the test fluids. Measured leak rates for these packages initially and after thermal shocking are given in Table 10. The leak rates of all packages, except the third  $6.9 \times 10^4 \text{ N/m}^2$  (10 psi) specimen which was found to have a gross leak at one of its leads, are within the allowed leak rate of  $5.7 \times 10^{-7} \text{ atm cc/sec}$  (air equivalent). This tended to substantiate the conjecture that seal failure of the initial packages during thermal shock was due to the fact that water was used as the test media. The results also indicate that somewhat lower and more consistent leak rates are obtained for a sealing pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi).

A similar set of 1.27 cm (1/2 inch) square packages was prepared and seal tested. These packages were thermal shocked between -65°C and +150°C using fluorocarbon as the test fluid. Results obtained are given in Table 11. As in the case of the 2.54 cm (1 inch) square packages, seal tests were performed after bombing the packages at 30 psig helium for three hours, and measured leak rates are given as air equivalents. The calculated permitted maximum leak rate for these packages (internal volume of 0.308 cc) is  $2.3 \times 10^{-6} \text{ atm cc/sec}$  (air equivalent). All the packages were well within this requirement. Also, these results indicate that while all of the sealing pressures are certainly adequate to give a good seal, the leak rate apparently decreases somewhat as the sealing pressure is increased. Since this result also is supported by the data obtained for the second set of 2.54 cm (1 inch)

Table 10. Seal Test Results (Initially and After Thermal Shocking) for Second Set of 2.54 cm (1 inch) Square Gold Plated Kovar Packages Sealed with 0.015 cm (6 mils) Thick Ablefilm 507 Preforms with Four Different Pressures Applied to the Seal Area

Sealing Pressure	Initial Measured Leak Rate Air Equivalent (atm cc/sec)	Measured Leak Rate Air Equivalent After Thermal Shocking 0°C to +100°C - 15 Cycles (atm cc/sec)
$3.5 \times 10^4 \text{ N/m}^2$ (5 psi)	$1.8 \times 10^{-7}$ $6.6 \times 10^{-8}$ $4.6 \times 10^{-8}$	$2.4 \times 10^{-7}$ $1.2 \times 10^{-8}$ $6.2 \times 10^{-8}$
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$6.4 \times 10^{-8}$ $4.4 \times 10^{-8}$ Gross Leak at Lead	$5.6 \times 10^{-8}$ $4.6 \times 10^{-8}$ ---
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$1.3 \times 10^{-7}$ $8.2 \times 10^{-8}$ $5.6 \times 10^{-8}$	$2.4 \times 10^{-7}$ $1.0 \times 10^{-7}$ $1.2 \times 10^{-7}$
$13.8 \times 10^4 \text{ N/m}^2$ (20 psi)	$5.8 \times 10^{-8}$ $4.2 \times 10^{-8}$ $5.4 \times 10^{-8}$	$1.2 \times 10^{-7}$ $1.2 \times 10^{-7}$ $1.5 \times 10^{-7}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $5.7 \times 10^{-7}$  atm cc/sec (air equivalent).

Table 11. Seal Test Results (Initially and After Thermal Shocking) for 1.27 cm (1/2 inch) Square Gold Plated Kovar Packages Sealed with 0.015 cm (6 mils) Thick Ablefilm 507 Preforms with Four Different Pressures Applied to the Seal Area

Sealing Pressure	Initial Measured Leak Rate Air Equivalent (atm cc/sec)	Measured Leak Rate Air Equivalent After Thermal Shocking -65°C to +150°C - 15 Cycles (atm cc/sec)
$3.5 \times 10^4 \text{ N/m}^2$ (5 psi)	$3.6 \times 10^{-8}$ $7.2 \times 10^{-8}$ $4.0 \times 10^{-8}$	$2.2 \times 10^{-8}$ $3.8 \times 10^{-8}$ $2.2 \times 10^{-8}$
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$4.0 \times 10^{-8}$ $4.4 \times 10^{-8}$ $2.5 \times 10^{-8}$	$3.0 \times 10^{-8}$ $2.5 \times 10^{-8}$ $1.2 \times 10^{-8}$
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$3.0 \times 10^{-8}$ $3.6 \times 10^{-8}$ $3.2 \times 10^{-8}$	$1.8 \times 10^{-8}$ $2.0 \times 10^{-8}$ $1.6 \times 10^{-8}$
$13.8 \times 10^4 \text{ N/m}^2$ (20 psi)	$1.4 \times 10^{-8}$ $1.2 \times 10^{-8}$ $2.8 \times 10^{-8}$	$8.6 \times 10^{-9}$ $7.2 \times 10^{-9}$ $1.6 \times 10^{-8}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $2.3 \times 10^{-6}$  atm cc/sec (air equivalent)

square packages, a sealing pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) will be used in all further work with film adhesives.

#### 2.3.2.4 Effect of Preform Thickness

Tests were performed to determine the effect of preform thickness on the quality of seals obtained using film adhesives. This information was desired so that an appropriate preform thickness could be selected for use in further tests comparing the film adhesives with each other and with the paste adhesive.

To investigate this effect, three additional packages of each size (2.54 cm square and 1.27 cm square) were sealed with an applied sealing pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) using 0.0076 and 0.020 cm (3 and 8 mils) thick Ablefilm 507 preforms. Results for these packages and for packages previously sealed with 0.015 cm (6 mils) thick preforms are given in Tables 12 and 13. Good seals were obtained in all cases except for one 2.54 cm package sealed with a 0.0076 cm (3 mils) thick preform. This package was a gross leaker. Data for the 2.54 cm packages (Table 12) indicate that the leak rate decreases as the preforms become successively thinner. Data for the 1.27 cm packages (Table 13) substantiates that the thicker preform (0.020 cm or 8 mils) gives the largest leak rate, but indicates that the leak rates for the 0.0076 and 0.015 cm (3 and 6 mils) thick preforms are very nearly the same. Based on this latter result and the fact that one of the 2.54 cm packages sealed with a 0.0076 cm (3 mils) thick preform was a gross leaker, it is felt that any improvement in leak rate that may be gained by using the 0.0076 cm (3 mils) thick preform is not worth the accompanying risk of obtaining gross leakers. It was concluded that for the present application (i.e., sealing a flat lid on a flat rimmed package), an appropriate preform thickness is 0.015 cm (6 mils).

#### 2.3.2.5 Comparison of Various Adhesives

Additional packages were sealed with the film adhesives Ablefilm 529 and Ablefilm 550 and the paste adhesive Epo-Tek H77. All packages sealed with film adhesives were sealed with 0.015 cm (6 mils) thick preforms with a pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) applied to the seal area during cure.

Table 12. Initial Seal Test Results for 2.54 cm (1 inch) Square Gold-Plated Kovar Packages Sealed With Ablefilm 507 Preforms of Three Different Thicknesses With a Pressure of  $13.8 \times 10^4$  N/m<sup>2</sup> (20 psi) Applied to the Seal Area

Preform Thickness	Initial Measured Leak Rate Air Equivalent (atm cc/sec)
0.0076 cm (3 mils)	$1.8 \times 10^{-8}$ Gross $1.4 \times 10^{-8}$
0.015 cm (6 mils)	$5.8 \times 10^{-8}$ $4.2 \times 10^{-8}$ $5.4 \times 10^{-8}$
0.020 cm (8 mils)	$1.2 \times 10^{-7}$ $1.4 \times 10^{-7}$ $2.6 \times 10^{-7}$

NOTE: MIL-STD-883A allowed maximum leak rate is  $5.7 \times 10^{-7}$  atm cc/sec (air equivalent)

**Table 13. Initial Seal Test Results for 1.27 cm (1/2 inch)  
Square Gold Plated Kovar Packages Sealed With  
Ablefilm 507 Preforms of Three Different Thicknesses  
With a Pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) applied  
to the Seal Area**

Preform Thickness	Initial Measured Leak Rate Air Equivalent (atm cc/sec)
0.0076 cm (3 mils)	$2.6 \times 10^{-8}$ $2.0 \times 10^{-8}$ $2.0 \times 10^{-8}$
0.015 cm (6 mils)	$1.4 \times 10^{-8}$ $1.2 \times 10^{-8}$ $2.8 \times 10^{-8}$
0.020 cm (8 mils)	$1.6 \times 10^{-7}$ $2.5 \times 10^{-7}$ $2.0 \times 10^{-7}$

**Note: MIL-STD-883A Allowed Maximum Leak Rate is  
 $2.3 \times 10^{-6}$  atm cc/sec (air equivalent)**

Packages sealed with the paste adhesive were clamped at a pressure of only  $3.5 \times 10^4 \text{ N/m}^2$  (5 psi) during cure. Ablefilm 529 and 550 were cured for two hours at 150°C and Epo-Tek H77 was cured for 30 minutes at 150°C.

Results for these packages and those obtained previously for Ablefilm 507 are given in Tables 14 and 15, respectively, for the 2.54 and 1.27 cm square packages. Ablefilm 529 preforms were available only for the 2.54 cm (1 inch) square packages. All packages had leak rates well below the permitted maximums. Data for the 1.27 cm square packages indicate that the leak rates for packages sealed with Ablefilm 507 and Ablefilm 550 are essentially the same. Data for the 2.54 cm square packages indicate that Ablefilm 550 may give a better seal than either Ablefilm 507 or Ablefilm 529. In both cases the data indicate that the paste adhesive Epo-Tek H77 gives seals with leak rates almost an order of magnitude less than those obtained with any of the film adhesives.

#### 2.3.2.6 Results of MIL-STD-883A, Class A Screen Tests

All packages were sequentially subjected to MIL-STD-883A, Class A Thermal Shock, Temperature Cycling, Mechanical Shock, and Constant Acceleration test environments. Seal integrity was determined initially, and after the packages were subjected to each test environment. Results are given in Tables 16 through 18 for the 2.54 cm (1 inch) square packages and in Tables 19 through 21 for the 1.27 cm (1/2 inch) square packages.

##### 2.3.2.6.1 Thermal Shock and Temperature Cycling Results

As shown in Tables 16 through 21, the measured leak rates of all packages are still less than the permitted maximum leak rates calculated from the equation given in Method 1014.1 of MIL-STD-883A ( $5.7 \times 10^{-7}$  atm cc/sec for the 2.54 cm square packages and  $2.3 \times 10^{-6}$  atm cc/sec for the 1.27 cm square packages) after exposure to the thermal shock and temperature cycling test environments. Thus, the data indicate that these environments did not degrade the package seals.

Table 14. Initial Seal Test Results for 54 cm (1 inch)  
Square Gold Plated Kovar Packages Sealed  
With Various Adhesives

Adhesive	Initial Measured Leak Rate Air Equivalent (atm cc/sec)
Ablefilm 507	$5.8 \times 10^{-8}$
	$4.2 \times 10^{-8}$
	$5.4 \times 10^{-8}$
Ablefilm 529	$1.0 \times 10^{-7}$
	$1.2 \times 10^{-7}*$
	$1.4 \times 10^{-7}$
	$1.2 \times 10^{-7}$
Ablefilm 550	$2.4 \times 10^{-8}$
	$1.5 \times 10^{-8}$
	$3.0 \times 10^{-8}$
Epo-Tek H77	$6.2 \times 10^{-9}$
	$4.4 \times 10^{-9}$
	$4.5 \times 10^{-9}$

Note: All packages sealed with film adhesives were sealed with 0.015 cm (6 mils) thick preforms with a pressure of  $13.8 \times 10^4$  N/m<sup>2</sup> (20 psi) applied to the seal area during cure. Packages sealed with the paste adhesive (Epo-Tek H77) were clamped at a pressure of only  $3.5 \times 10^4$  N/m<sup>2</sup> (5 psi) during cure.

\* The lid on this package slipped during cure and is bonded to only approximately half the seal area of the package rim on two sides.

Table 15. Initial Seal Test Results for 1.27 cm (1/2 inch)  
Square Gold Plated Kovar Packages Sealed With  
Various Adhesives

Adhesive	Initial Measured Leak Rate Air Equivalent (atm cc/sec)
Ablefilm 507	$1.4 \times 10^{-8}$ $1.2 \times 10^{-8}$ $2.8 \times 10^{-8}$
Ablefilm 550	$2.4 \times 10^{-8}$ $1.6 \times 10^{-8}$ $1.4 \times 10^{-8}$
Epo-Tek H77	$2.8 \times 10^{-9}$ $1.0 \times 10^{-9}$ $1.4 \times 10^{-9}$

Note: All packages sealed with film adhesives were sealed with 0.015 cm (6 mils) thick preforms with a pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) applied to the seal area during cure. Packages sealed with the paste adhesive (Epo-Tek H77) were clamped at a pressure of only  $3.5 \times 10^4 \text{ N/m}^2$  (5 psi) during cure.

Table 16. Seal Test Results for 2.54 cm (1 inch) Square Gold-Plated Kovar Packages Sealed With 0.015 cm (6 mils) Thick Ablefilm 507 Preforms With Four Different Pressures Applied to the Seal Area

Sealing Pressure	Initial	Measured Leak Rate (Air Equivalent)			After Mechanical Shock 1500 g's 5 Shocks	After Constant Acc. 10,000 g's
		After Thermal Shock 0°C to +100°C 15 Cycles	After Temp. Cycling -65°C to +150°C 15 Cycles	After Mounting on Tabs		
$3.5 \times 10^4 \text{ N/m}^2$ (5 psi)	$1.8 \times 10^{-7}$	$2.4 \times 10^{-7}$	$2.0 \times 10^{-7}$	$6.0 \times 10^{-7}$	$5.8 \times 10^{-7}$	Lid Came Off
	$6.6 \times 10^{-8}$	$1.2 \times 10^{-7}$	$1.4 \times 10^{-7}$	$1.2 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross**
	$4.6 \times 10^{-8}$	$6.2 \times 10^{-8}$	$6.4 \times 10^{-8}$	$3.1 \times 10^{-7}$	$2.4 \times 10^{-7}$	$4.2 \times 10^{-7}$
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$6.4 \times 10^{-8}$	$5.6 \times 10^{-8}$	$1.0 \times 10^{-7}$	$5.1 \times 10^{-7}$	$3.2 \times 10^{-7}$	$5.2 \times 10^{-7}$
	$4.4 \times 10^{-8}$	$4.6 \times 10^{-8}$	$6.2 \times 10^{-8}$	$4.6 \times 10^{-7}$	*	Lid Came Off
	Gross Leak at Lead	--	--	--	--	--
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$1.3 \times 10^{-7}$	$2.4 \times 10^{-7}$	$1.4 \times 10^{-7}$	$6.2 \times 10^{-7}$	$6.2 \times 10^{-7}$	Gross**
	$8.2 \times 10^{-8}$	$1.0 \times 10^{-7}$	$4.8 \times 10^{-8}$	$3.8 \times 10^{-7}$	$3.6 \times 10^{-7}$	$3.6 \times 10^{-7}$
	$5.6 \times 10^{-8}$	$1.2 \times 10^{-7}$	$5.6 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.8 \times 10^{-7}$	Gross**
$13.8 \times 10^4 \text{ N/m}^2$ (20 psi)	$5.8 \times 10^{-8}$	$1.2 \times 10^{-7}$	$6.0 \times 10^{-8}$	$4.2 \times 10^{-7}$	$3.9 \times 10^{-7}$	$3.4 \times 10^{-7}$
	$4.2 \times 10^{-8}$	$1.2 \times 10^{-7}$	$5.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross**
	$5.4 \times 10^{-8}$	$1.5 \times 10^{-7}$	$7.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross

\*Could not get fine leak reading, but gross leak test did not indicate package to be a gross leaker.

\*\*Package came off mounting tab.

Table 17. Seal Test Results for 2.54 cm (1 inch) Square Gold-Plated Kovar Packages Sealed With Ablefilm 507 Preforms of Three Different Thicknesses With a Pressure of  $13.8 \times 10^4$  N/m<sup>2</sup> (20 psi) Applied to the Seal Area

Preform Thickness	Measured Leak Rate (Air Equivalent) (atm cc/sec)				
	Initial	After Thermal Shock 0°C to +100°C 15 Cycles	After Temp. Cycling -65°C to +150°C 15 Cycles	After Mounting on Tabs	After Mechanical Shock 1500 g's 5 Shocks
0.0076 cm (3 mils)	$1.8 \times 10^{-8}$	$3.8 \times 10^{-8}$	$3.4 \times 10^{-8}$	$2.2 \times 10^{-7}$	$2.0 \times 10^{-7}$
	Gross	--	--	--	--
0.015 cm (6 mils)	$1.4 \times 10^{-8}$	$1.6 \times 10^{-8}$	$2.2 \times 10^{-8}$	$2.7 \times 10^{-7}$	$2.4 \times 10^{-7}$
	$5.8 \times 10^{-8}$	$1.2 \times 10^{-7}$	$6.0 \times 10^{-8}$	$4.2 \times 10^{-7}$	$3.9 \times 10^{-7}$
0.020 cm (8 mils)	$4.2 \times 10^{-8}$	$1.2 \times 10^{-7}$	$5.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$
	$5.4 \times 10^{-8}$	$1.5 \times 10^{-7}$	$7.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$
	$1.2 \times 10^{-7}$	$2.2 \times 10^{-7}$	$9.6 \times 10^{-8}$	$4.4 \times 10^{-7}$	$4.0 \times 10^{-7}$
	$1.4 \times 10^{-7}$	$2.0 \times 10^{-7}$	$1.2 \times 10^{-7}$	$4.0 \times 10^{-7}$	$3.6 \times 10^{-7}$
	$2.6 \times 10^{-7}$	$3.0 \times 10^{-7}$	$1.6 \times 10^{-7}$	$6.2 \times 10^{-7}$	$5.8 \times 10^{-7}$

\*\* Package Came Off Mounting Tab

Table 18. Seal Test Results for 2.54 cm (1 inch) Square Gold-Plated Kovar Packages Sealed With Various Adhesives and Solder.

		Measured Leak Rate (Air Equivalent) (atm cc/sec)					
Adhesive	Initial	After Thermal Shock 0°C to +100°C 15 Cycles	After Temp. Cycling -65°C to +150°C 15 Cycles	After Mounting on Tabs	After Mechanical Shock 1500 g's 5 Shocks	After Constant Acc. 10,000 g's	
		$5.8 \times 10^{-8}$	$1.2 \times 10^{-7}$	$6.0 \times 10^{-8}$	$4.2 \times 10^{-7}$	$3.9 \times 10^{-7}$	$3.4 \times 10^{-7}$
Ablefilm 507	$4.2 \times 10^{-8}$	$1.2 \times 10^{-7}$	$5.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross**	
	$5.4 \times 10^{-8}$	$1.5 \times 10^{-7}$	$7.8 \times 10^{-8}$	$4.0 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross	
	$1.0 \times 10^{-7}$	$7.0 \times 10^{-8}$	$7.6 \times 10^{-8}$	$2.0 \times 10^{-7}$	$2.4 \times 10^{-7}$	Lid came off	
	$1.2 \times 10^{-7}*$	$8.8 \times 10^{-8}$	$8.8 \times 10^{-8}$	$4.2 \times 10^{-7}$	$2.6 \times 10^{-7}$	Lid came off	
Ablefilm 529	$1.4 \times 10^{-7}$	$7.8 \times 10^{-8}$	$9.2 \times 10^{-8}$	$2.9 \times 10^{-7}$	$3.0 \times 10^{-7}$	Lid came off	
	$1.2 \times 10^{-7}$	$7.2 \times 10^{-8}$	$7.6 \times 10^{-8}$	$2.4 \times 10^{-7}$	$2.4 \times 10^{-7}$	Lid came off	
	$2.4 \times 10^{-8}$	$7.4 \times 10^{-9}$	$1.0 \times 10^{-8}$	$1.8 \times 10^{-7}$	$2.0 \times 10^{-7}$	$3.2 \times 10^{-7}$	
	$1.5 \times 10^{-8}$	$8.2 \times 10^{-9}$	$1.0 \times 10^{-8}$	$1.9 \times 10^{-7}$	$2.1 \times 10^{-7}$	Gross**	
Ablefilm 550	$3.0 \times 10^{-8}$	$7.6 \times 10^{-9}$	$8.8 \times 10^{-9}$	$1.3 \times 10^{-7}$	$1.3 \times 10^{-7}$	Gross**	
	$2.4 \times 10^{-8}$	$7.4 \times 10^{-9}$	$1.0 \times 10^{-8}$	$1.8 \times 10^{-7}$	$2.0 \times 10^{-7}$	Lid came off	
	$1.5 \times 10^{-8}$	$8.2 \times 10^{-9}$	$1.0 \times 10^{-8}$	$1.9 \times 10^{-7}$	$2.1 \times 10^{-7}$	Lid came off	
	$3.0 \times 10^{-8}$	$7.6 \times 10^{-9}$	$8.8 \times 10^{-9}$	$1.3 \times 10^{-7}$	$1.3 \times 10^{-7}$	Lid came off	
Epo-Tek H77	$6.2 \times 10^{-9}$	$2.8 \times 10^{-8}$	$2.0 \times 10^{-8}$	$4.2 \times 10^{-7}$	$2.6 \times 10^{-7}$	Lid came off	
	$4.4 \times 10^{-9}$	$1.8 \times 10^{-8}$	$1.6 \times 10^{-8}$	$4.1 \times 10^{-7}$	$2.6 \times 10^{-7}$	Lid came off	
	$4.5 \times 10^{-9}$	$1.2 \times 10^{-8}$	$1.2 \times 10^{-8}$	$3.6 \times 10^{-7}$	$2.0 \times 10^{-7}$	Lid came off	
	$4.0 \times 10^{-9}$	Not measured	Not measured	$3.2 \times 10^{-7}$	$2.0 \times 10^{-7}$	Gross	
Solder	$3.6 \times 10^{-9}$	Not measured	Not measured	$2.7 \times 10^{-7}$	$2.0 \times 10^{-7}$	Lid came off	

NOTE: All packages sealed with film adhesives were sealed with 0.015 cm (6 mils) thick preforms with a pressure of  $13.8 \times 10^4$  N/m<sup>2</sup> (20 psi) applied to the seal area during cure. Packages sealed with the paste adhesive (Epo-Tek H77) were clamped at a pressure of only  $3.5 \times 10^4$  N/m<sup>2</sup> (5 psi) during cure.

\* The lid on this package slipped during cure and is bonded to only approximately half the seal area of the package rim on two sides.

\*\* Package came off mounting tab.

Table 19. Seal Test Results for 1.27 cm (1/2 inch) Square Gold-Plated Kovar Packages Sealed With 0.015 cm (6 mils) Thick Ablefilm 507 Preforms With Four Different Pressures Applied to the Seal Area

		Measured Leak Rate (Air Equivalent) (atm cc/sec)					
Sealing Pressure	Initial	After Thermal Shock -65°C to +150°C 15 Cycles		After Temp. Cycling -65°C to +150°C 15 Cycles		After Mechanical Shock 1500 g's 5 Shocks	After Constant Acc. 10,000 g's
		After Thermal Shock -65°C to +150°C 15 Cycles	After Mounting on Tabs	After Mounting on Tabs	After 1500 g's 5 Shocks		
$3.5 \times 10^4 \text{ N/m}^2$ (5 psi)	$3.6 \times 10^{-8}$	$2.2 \times 10^{-8}$	$3.8 \times 10^{-8}$	$1.4 \times 10^{-7}$	$9.6 \times 10^{-8}$	$1.0 \times 10^{-7}$	
	$7.2 \times 10^{-8}$	$3.8 \times 10^{-8}$	$8.0 \times 10^{-8}$	$2.4 \times 10^{-7}$	$1.6 \times 10^{-7}$	$1.7 \times 10^{-7}$	
	$4.0 \times 10^{-8}$	$2.2 \times 10^{-8}$	$4.2 \times 10^{-8}$	$1.9 \times 10^{-7}$	$1.2 \times 10^{-7}$	$1.4 \times 10^{-7}$	
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$4.0 \times 10^{-8}$	$3.0 \times 10^{-8}$	$4.6 \times 10^{-8}$	$1.8 \times 10^{-7}$	$1.5 \times 10^{-7}$	$1.7 \times 10^{-7}$	
	$4.4 \times 10^{-8}$	$2.5 \times 10^{-8}$	$4.4 \times 10^{-8}$	$1.6 \times 10^{-7}$	$1.4 \times 10^{-7}$	$1.6 \times 10^{-7}$	
	$2.5 \times 10^{-8}$	$1.2 \times 10^{-8}$	$3.0 \times 10^{-8}$	$1.3 \times 10^{-7}$	$1.0 \times 10^{-7}$	$1.4 \times 10^{-7}$	
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$3.0 \times 10^{-8}$	$1.8 \times 10^{-8}$	$3.4 \times 10^{-8}$	$1.9 \times 10^{-7}$	$1.6 \times 10^{-7}$	$1.1 \times 10^{-7}$	
	$3.6 \times 10^{-8}$	$2.0 \times 10^{-8}$	$3.8 \times 10^{-8}$	$1.5 \times 10^{-7}$	$1.2 \times 10^{-7}$	$1.0 \times 10^{-7}$	
	$3.2 \times 10^{-8}$	$1.6 \times 10^{-8}$	$3.6 \times 10^{-8}$	$1.8 \times 10^{-7}$	$1.4 \times 10^{-7}$	$1.1 \times 10^{-7}$	
$13.8 \times 10^4 \text{ N/m}^2$ (20 psi)	$1.4 \times 10^{-8}$	$8.6 \times 10^{-9}$	$1.6 \times 10^{-8}$	$1.2 \times 10^{-7}$	$1.0 \times 10^{-7}$	$7.4 \times 10^{-8}$	
	$1.2 \times 10^{-8}$	$7.2 \times 10^{-9}$	$1.3 \times 10^{-8}$	$1.0 \times 10^{-7}$	$7.8 \times 10^{-8}$	$7.8 \times 10^{-8}$	
	$2.8 \times 10^{-8}$	$1.6 \times 10^{-8}$	$3.2 \times 10^{-8}$	$1.5 \times 10^{-7}$	$1.2 \times 10^{-7}$	$1.0 \times 10^{-7}$	

Table 20. Seal Test Results for 1.27 cm (1/2 inch) Square Gold-Plated Kovar Packages Sealed With Ablefilm 507 Preforms of Three Different Thicknesses With a Pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) Applied to the Seal Area

Preform Thickness	Measured Leak Rate (Air Equivalent) (atm cc/sec)					
	Initial	After Thermal Shock 0°C to +100°C 15 Cycles	After Temp. Cycling -65°C to +150°C 15 Cycles	After Mounting on Tabs	After Mechanical Shock 1500 g's 5 Shocks	After Constant Acc. 10,000 g's
0.0076 cm (3 mils)	$2.6 \times 10^{-8}$	$2.1 \times 10^{-8}$	$2.8 \times 10^{-8}$	$1.6 \times 10^{-7}$	$9.0 \times 10^{-8}$	$3.2 \times 10^{-7}$
	$2.0 \times 10^{-8}$	$1.4 \times 10^{-8}$	$2.4 \times 10^{-8}$	$1.6 \times 10^{-7}$	$9.2 \times 10^{-8}$	$3.4 \times 10^{-7}$
	$2.0 \times 10^{-8}$	$1.6 \times 10^{-8}$	$2.2 \times 10^{-8}$	$1.3 \times 10^{-7}$	$7.4 \times 10^{-8}$	$8.2 \times 10^{-8}$
0.015 cm* (6 mils)	$1.4 \times 10^{-8}$	$8.6 \times 10^{-9}$	$1.6 \times 10^{-8}$	$1.2 \times 10^{-7}$	$1.0 \times 10^{-7}$	$7.4 \times 10^{-8}$
	$1.2 \times 10^{-8}$	$7.2 \times 10^{-9}$	$1.3 \times 10^{-8}$	$1.0 \times 10^{-7}$	$7.8 \times 10^{-8}$	$7.8 \times 10^{-8}$
	$2.8 \times 10^{-8}$	$1.6 \times 10^{-8}$	$3.2 \times 10^{-8}$	$1.5 \times 10^{-7}$	$1.2 \times 10^{-7}$	$1.0 \times 10^{-7}$
0.020 cm (8 mils)	$1.6 \times 10^{-7}$	$1.6 \times 10^{-7}$	$1.4 \times 10^{-7}$	$2.4 \times 10^{-7}$	$2.2 \times 10^{-7}$	$2.4 \times 10^{-7}$
	$2.5 \times 10^{-7}$	$1.6 \times 10^{-7}$	$1.3 \times 10^{-7}$	$2.5 \times 10^{-7}$	$2.1 \times 10^{-7}$	$2.5 \times 10^{-7}$
	$2.0 \times 10^{-7}$	$1.2 \times 10^{-7}$	$1.4 \times 10^{-7}$	$3.2 \times 10^{-7}$	Gross	-

\*These packages were thermal shocked between -65°C and +150°C for 15 cycles.

Table 21. Seal Test Results for 1.27 cm (1/2 inch) Square Gold-Plated Kovar Packages Sealed With Various Adhesives

Adhesive	Measured Leak Rate (Air Equivalent) (atm cc/sec)					
	Initial	After Thermal Shock 0°C to +100°C 15 Cycles	After Temp. Cycling -65°C to +150°C 15 Cycles	After Mounting on Tabs	After Mechanical Shock 1500 g's 5 Shocks	After Constant Acc. 10,000 g's
Ablefilm 507*	1.4 x 10 <sup>-8</sup>	8.6 x 10 <sup>-9</sup>	1.6 x 10 <sup>-8</sup>	1.2 x 10 <sup>-7</sup>	1.0 x 10 <sup>-7</sup>	7.4 x 10 <sup>-8</sup>
	1.2 x 10 <sup>-8</sup>	7.2 x 10 <sup>-9</sup>	1.3 x 10 <sup>-8</sup>	1.0 x 10 <sup>-7</sup>	7.8 x 10 <sup>-8</sup>	7.8 x 10 <sup>-8</sup>
	2.8 x 10 <sup>-8</sup>	1.6 x 10 <sup>-8</sup>	3.2 x 10 <sup>-8</sup>	1.5 x 10 <sup>-7</sup>	1.2 x 10 <sup>-7</sup>	1.0 x 10 <sup>-7</sup>
Ablefilm 550	2.4 x 10 <sup>-8</sup>	1.6 x 10 <sup>-8</sup>	1.8 x 10 <sup>-8</sup>	1.6 x 10 <sup>-7</sup>	1.0 x 10 <sup>-7</sup>	8.8 x 10 <sup>-8</sup>
	1.6 x 10 <sup>-8</sup>	1.5 x 10 <sup>-8</sup>	6.4 x 10 <sup>-9</sup>	1.4 x 10 <sup>-7</sup>	8.2 x 10 <sup>-8</sup>	6.8 x 10 <sup>-8</sup>
	1.4 x 10 <sup>-8</sup>	1.4 x 10 <sup>-8</sup>	1.4 x 10 <sup>-8</sup>	1.0 x 10 <sup>-7</sup>	6.0 x 10 <sup>-8</sup>	5.4 x 10 <sup>-8</sup>
Epo-Tek H77*	2.8 x 10 <sup>-9</sup>	1.4 x 10 <sup>-9</sup>	4.2 x 10 <sup>-9</sup>	1.5 x 10 <sup>-7</sup>	1.1 x 10 <sup>-7</sup>	1.0 x 10 <sup>-7</sup>
	1.0 x 10 <sup>-9</sup>	0.5 x 10 <sup>-9</sup>	2.4 x 10 <sup>-9</sup>	8.2 x 10 <sup>-8</sup>	6.2 x 10 <sup>-8</sup>	5.2 x 10 <sup>-8</sup>
	1.4 x 10 <sup>-9</sup>	0.4 x 10 <sup>-9</sup>	2.8 x 10 <sup>-9</sup>	6.4 x 10 <sup>-8</sup>	3.8 x 10 <sup>-8</sup>	3.4 x 10 <sup>-8</sup>

NOTE: All packages sealed with film adhesives were sealed with 0.015 cm (6 mils) thick preforms with a pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) applied to the seal area during cure. Packages sealed with the paste adhesive (Epo-Tek H77) were clamped at a pressure of only  $3.5 \times 10^4 \text{ N/m}^2$  (5 psi) during cure.

\* These packages were thermal shocked between -65°C and +150°C for 15 cycles.

### 2.3.2.6.2 Method of Mounting Packages for Mechanical Shock and Constant Acceleration Testing

In order to perform the mechanical shock and constant acceleration tests, the packages were mounted on tabs. Aluminum tabs 4.28 cm (1.68 inches) long by 3.65 cm (1.43 inches) wide were fabricated for this purpose. This size tab was selected so that it could be used with either the 2.54 cm (1 inch) square or the 1.27 cm (1/2 inch) square packages. The tabs were made 0.40 cm (5/32 inch) thick so that they would not flex at high "g" loading. Photographs of packages mounted on tabs are shown in Figures 2 and 3. Photographs of the test fixtures for shock and constant acceleration testing are shown in Figures 4 and 6, respectively; and a photograph showing how the test specimens were oriented in the shock fixture is given in Figure 5.

Preliminary tests using four reject packages were run to select a suitable adhesive for mounting the packages on the aluminum tabs. Initially an adhesive was tested which cures quickly at room temperature and is reported to have very high tensile strength. However, it failed (i.e., all four packages mounted with it came off the aluminum tabs) at less than 10,000 g's centrifuge or at a pressure of approximately  $8.8 \times 10^5 \text{ N/m}^2$  (125 psi). While the reason for failure of this adhesive is not known, it is felt that it was due to the shear force caused by the sudden acceleration of the centrifuge rotor from 0 to 10,000 g's rather than the tensile force established at 10,000 g's. Epo-Tek H77 was then tested and proved successful, so this adhesive was selected as the mounting adhesive.

The packages to be tested were mounted on the aluminum tabs with this adhesive, clamped in position, and the adhesive was cured at 130°C for one hour. Various clamping pressures were used so that this parameter could be evaluated for future reference. Since this same mounting technique will be used in the follow-on study, this opportunity was taken to see if the ability of the bond to withstand 10,000 g's centrifuge depended on the clamping pressure applied during cure. Eight of the twenty-eight 2.54 cm (1 inch) square packages tested came loose from their aluminum tabs during centrifuging, however there was no correlation of adhesive failure with clamping

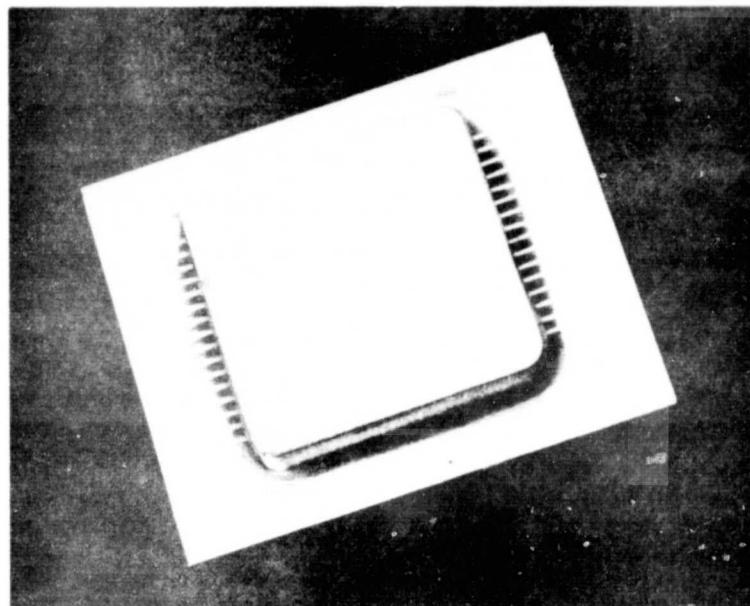


Figure 2. Aluminum Tab With 2.54 cm (1 inch) Square Package Mounted on it.

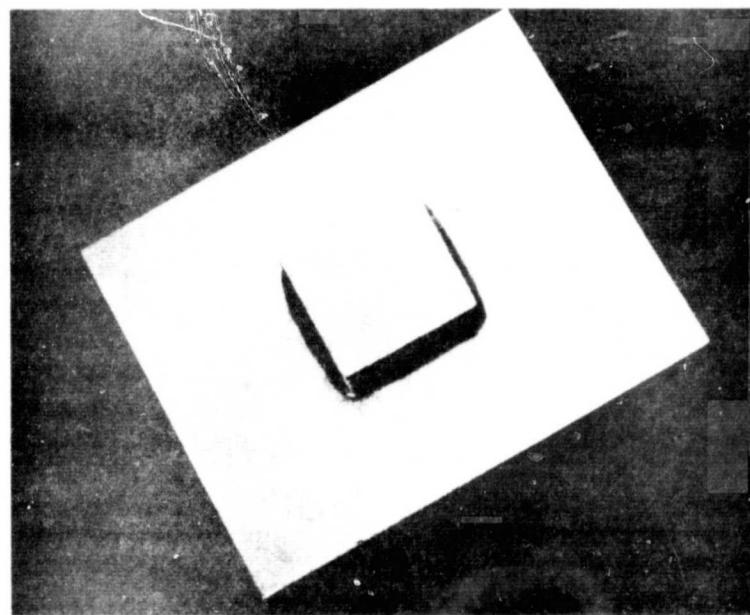


Figure 3. Aluminum Tab With 1.27 cm (1/2 inch) Square Package Mounted on it

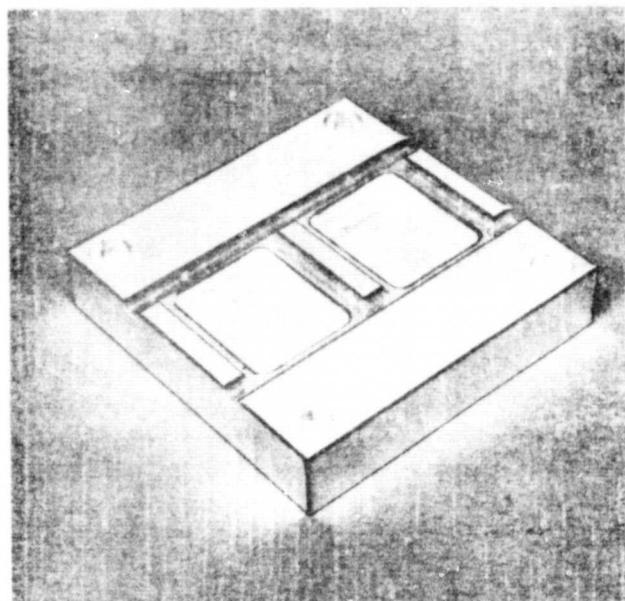


Figure 4. Test Fixture for Shock Testing

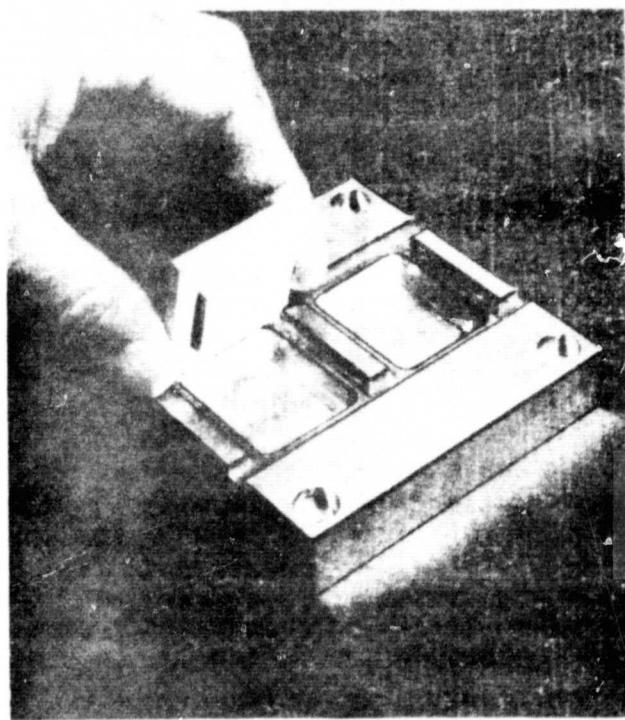


Figure 5. Shock Test Fixture and Specimen to be Tested.  
Specimen is Placed in Fixture with Package Down.

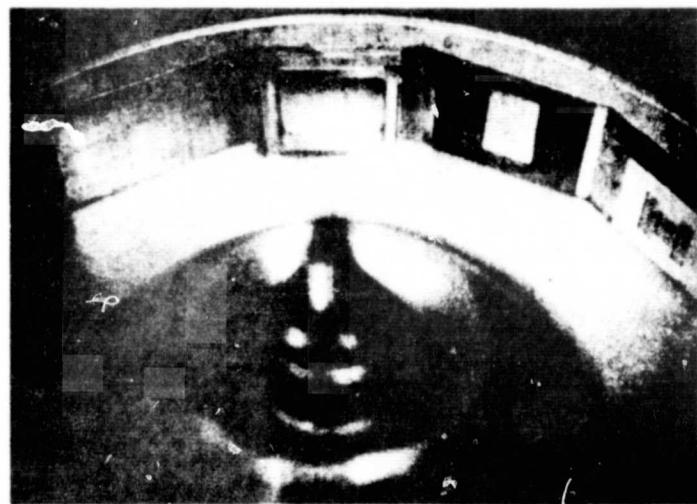


Figure 6. Centrifuge Ring Showing Fixture in Which Package is Mounted. Specimen is Placed in Fixture With Package Pointed Outward From Center of Centrifuge

pressure for the range of clamping pressures used. These failures indicate that Epo-Tek H77 is marginal for this application and that other adhesives must be tested to select one for use in the follow-on study.

#### 2.3.2.6.3 Effect of Adhesively Mounting Packages on Tabs

As shown in Tables 16 through 21, seal tests also were run after the packages were mounted on the aluminum tabs. This was done for two reasons; (1) to determine if subjection of the packages to elevated temperature with a clamping pressure applied had affected the quality of their seals; and, (2) to determine if the presence of the additional epoxy adhesive had increased the apparent values of the fine leak rates due to its adsorption or absorption of helium during helium bombing and subsequent release during fine leak testing. This latter situation is possible since the exposed surface area of the adhesive used for mounting the packages is relatively large (of the order of 1 cm<sup>2</sup>). It appears that this effect occurred, resulting in an increase of apparent leak rate ranging up to approximately an order of magnitude for both sizes of packages sealed with epoxy adhesives other than Epo-Tek H77, and a factor of 20 to 40 for packages sealed with Epo-Tek H77. Also, for the two solder sealed 2.54 cm (1 inch) square packages, the measured leak rate increased by approximately two orders of magnitude. It should be noted that all leak rates are now in the low to middle 10<sup>-7</sup> atm cc/sec (air equivalent) range. These facts indicate that the leak rates being measured are not indicative of the true leak rates of the packages, but are indicative of the release of the helium adsorbed or absorbed by the adhesive (Epo-Tek H77) used to mount the packages. The helium being released is equivalent to a leak rate in the low 10<sup>-7</sup> atm cc/sec range and is obscuring the true leak rates of the packages which are less than this.

Seal integrity also was tested for the 2.54 cm (1 inch) square packages after the leads were trimmed and prior to mounting them on the aluminum tabs. Results showed that trimming the leads did not adversely affect the integrity of the packages.

#### 2.3.2.6.4 Mechanical Shock Test Results

Subjecting the packages to mechanical shock caused failure of one of the 1.27 cm (1/2 inch) square packages sealed with a 0.020 cm (8 mils) thick Ablefilm 507 preform and one of the 2.54 cm (1 inch) square packages sealed with a 0.015 cm (6 mils) thick Ablefilm 507 preform. The 1.27 cm (1/2 inch) square package was definitely a gross leaker; however, while a fine leak reading could not be obtained for the 2.54 cm (1 inch) square package, gross leak testing also did not indicate that it was a gross leaker. One of the two 2.54 cm (1 inch) square solder sealed packages, which were introduced at this point as controls, also failed (became a gross leaker) during mechanical shock testing.

#### 2.3.2.6.5 Constant Acceleration Test Results

The 10,000 g's constant acceleration stress was found to be a very severe test, not only for the epoxy adhesive sealed packages but also for the solder sealed controls. Only six of the twenty-six 2.54 cm (1 inch) square packages sealed with adhesives retained their seal integrity. All others, including the two solder sealed controls, failed. A summary of the results obtained is given in Table 22. The lids came off ten of the packages and twelve other packages were found to be gross leakers.

The conduct of this test requires that the chamber be evacuated to minimize drag for efficient operation of the centrifuge. As a result, the packages are subjected to a differential pressure of one atmosphere in addition to the 10,000 g's force due to centrifuging. The force due to the pressure differential also is acting outward against the lid, increasing the severity of the test. Consideration should be given to reducing this requirement (possibly to 5,000 g's) for packages of this size (2.54 cm or one inch square).

In contrast to these results, all the 1.27 cm (1/2 inch) square packages passed the 10,000 g's constant acceleration test. A comparison of the packages indicates two factors which can account for this. The first is that the smaller lid (1.27 cm or 1/2 inch square) has a larger seal area to total lid area ratio than the larger lid (2.54 cm or 1 inch square). This ratio

Table 22. Summary of Results for 2.54 cm (1 inch) Square Gold Plated Kovar Packages After Being Subjected to 10,000 g's Constant Acceleration

Adhesive	Preform Thickness	Seal Pressure	No. of Packages Tested	Retained Seal Integrity	Gross Leaker	Lid Came Off
Ablefilm 507	0.015 cm (6 mils)	$3.5 \times 10^4$ N/m <sup>2</sup> (5 psi)	3	1	1	1
Ablefilm 507	0.015 cm (6 mils)	$6.9 \times 10^4$ N/m <sup>2</sup> (10 psi)	2	1	--	1
Ablefilm 507	0.015 cm (6 mils)	$10.3 \times 10^4$ N/m <sup>2</sup> (15 psi)	3	1	2	--
Ablefilm 507	0.015 cm (6 mils)	$13.8 \times 10^4$ N/m <sup>2</sup> (20 psi)	3	1	2	--
Epo-Tek H77	N/A	$3.5 \times 10^4$ N/m <sup>2</sup> (5 psi)	3	--	--	3
Ablefilm 507	0.0076 cm (3 mils)	$13.8 \times 10^4$ N/m <sup>2</sup> (20 psi)	2	--	2	--
Ablefilm 507	0.020 cm (8 mils)	$13.8 \times 10^4$ N/m <sup>2</sup> (20 psi)	3	1	2	--
Ablefilm 529	0.015 cm (6 mils)	$13.8 \times 10^4$ N/m <sup>2</sup> (20 psi)	4	--	--	4
Ablefilm 550	0.015 cm (6 mils)	$13.8 \times 10^4$ N/m <sup>2</sup> (20 psi)	3	1	2	--
Solder		N/A	2	--	1	1

is 0.055/0.25 or 0.22 for the smaller packages and 0.16 for the larger packages. The second is that the smaller lid will have less tendency to flex when stressed than the larger lid. Apparently the combination of these factors is adequate to make the difference between essentially total failure for the 2.54 cm (1 inch) square packages and complete success for the 1.27 cm (1/2 inch) square packages.

The usual method of mounting packages for shock and constant acceleration testing is with the lid supported against the test fixtures since it is the adherence of the devices and/or substrate that is being tested. In the present case, the packages were mounted with the lid unsupported since the adherence of the lid was being tested. Consequently there is no other data with which to compare these results and it is not known whether or not seal failure should be expected for the larger packages when they are centrifuged at 10,000 g's. However, it is interesting to note that while twenty of the twenty-six 2.54 cm (1 inch) square packages sealed with the adhesives failed, both of the solder sealed packages also failed. This indicates that one type of metallurgically sealed package fares no better than epoxy adhesive sealed packages under this test condition.

#### 2.3.2.7 Epoxy Sealing of 5.08 cm (2 inch) Square Packages

Attempts were made to seal 5.08 cm (2 inch) square packages. Initially, three packages were sealed using the paste adhesive, Epo-Tek H77, with a pressure of approximately  $5.2 \times 10^4 \text{ N/m}^2$  (7.5 psi) applied to the seal area during cure. The packages were cured for 30 minutes at 150°C. Seal testing after bombing for three hours at 30 psig showed all three packages to be gross leakers. Next, three additional packages were sealed with Epo-Tek H77 (two with a pressure of  $10.3 \times 10^4 \text{ N/m}^2$  or 15 psi and one with  $13.8 \times 10^4 \text{ N/m}^2$  or 20 psi applied) and again cured for 30 minutes at 150°C. Also, three packages were sealed using 0.015 cm (6 mils) thick preforms of Ablefilm 550 with a pressure of  $13.8 \times 10^4 \text{ N/m}^2$  (20 psi) applied to the seal area during cure. These packages were cured for two hours at 150°C. Seal testing, after bombing for three hours at 30 psig, showed all six packages to be gross leakers.

It was speculated that the inability to obtain good seals might be due to the fact that pressure was building up in the packages during cure and venting through the adhesive causing the formation of small holes. To avoid this situation, 0.040 cm (1/64 inch or approximately 16 mils) diameter holes were drilled in six lids and packages using these lids were sealed with Epo-Tek H77. Three of these packages were sealed with zero pressure applied during cure and three were sealed with approximately  $3.5 \times 10^4 \text{ N/m}^2$  (5 psi) applied. All packages were cured at 150°C for 30 minutes. After curing, the vent or breather holes were hand sealed with solder. Again, seal testing showed all six packages to be gross leakers; so apparently, pressure build-up and venting during cure is not the responsible failure mechanism.

At this point, it was suggested that since these packages have a large surface area (25.8 sq. cm or 4 sq. inches) and a relatively narrow (0.102 cm or 40 mils) seal rim, seal failure was being caused by the helium bombing conditions (30 psig for three hours). To test this assumption, three packages were solder sealed and gross leak tested prior to bombing. All three packages passed this test (i.e., none were gross leakers). The packages were then bombed at 30 psig for three hours and retested for gross leak only. All were found to be gross leakers. The packages were reworked and gross leak tested to verify that the leaks had been repaired, and then subjected to the complete leak test procedure. Again, all three packages were found to be gross leakers. This substantiates that bombing these large packages at 30 psig is the cause of seal failure, and that even metallurgically sealed (solder sealed) packages fail under this condition. It is recommended that an investigation be made to determine a suitable bombing pressure and exposure time for seal testing packages of this size (5.08 cm or 2 inches square).

#### 2.3.2.8 Investigation of Removal and Replacement of Lids

Six packages that had been sealed with Ablefilm 507 were delidded and resealed using 0.020 cm (8 mils) thick Ablefilm 507 preforms. The packages were delidded by placing them on a hot plate at approximately 180 or 190°C and carefully prying the lids off with an X-Acto knife. The film adhesive was picked off with tweezers and the package rim and lids were recleaned.

A new preform was put in place, the packages were assembled according to the procedure previously described and then cured. Results obtained for these reworked packages are shown in Table 23. All reworked packages are well within the allowed maximum leak rate of  $5.7 \times 10^{-7}$  atm cc/sec (air equivalent). For this adhesive (Ablefilm 507), rework is simple and easily performed; however, the package must be subjected to an elevated temperature of at least 180°C for a few minutes for delidding.

Table 23. Seal Test Results for Reworked (Delidded and Resealed)  
 2.54 cm (1 inch) Square Gold Plated Kovar Packages  
 Sealed With 0.020 cm (8 mils) Thick Ablefilm 507 Preforms

Sealing Pressure	Measured Leak Rate Air Equivalent (atm cc/sec)
$6.9 \times 10^4 \text{ N/m}^2$ (10 psi)	$8.2 \times 10^{-8}$
	$1.2 \times 10^{-7}$
	$5.8 \times 10^{-8}$
$10.3 \times 10^4 \text{ N/m}^2$ (15 psi)	$1.0 \times 10^{-7}$
	$7.6 \times 10^{-8}$
	$1.0 \times 10^{-7}$

### 3.0 CONCLUSIONS AND RECOMMENDED FOLLOW-ON PROGRAM

This investigation has demonstrated that adhesive sealed packages retain their seal integrity (as determined by the seal test specified in MIL-STD-883A, Method 1014.1, Test Conditions A<sub>2</sub> and C<sub>1</sub>) after they are sequentially subjected to MIL-STD-883A, Class A Thermal Shock, Temperature Cycling, Mechanical Shock and Constant Acceleration test environments. Specifically, 1.27 cm (1/2 inch) square gold-plated Kovar flatpack-type packages sealed with the film adhesives Ablefilm 507, 529 and 550 and the paste adhesive Epo-Tek H77 passed all tests. Also, similarly prepared 2.54 cm (1 inch) square packages of the same type passed all tests except the 10,000 g's constant acceleration test. Only six out of 26 of these packages passed; however, two out of two solder sealed packages of this size and type introduced as controls also failed the constant acceleration test. This indicates that perhaps the 10,000 g's test level is too severe for packages of this size (2.54 cm or 1 inch square) and that consideration should be given to reducing it (possibly to 5,000 g's).

These results are encouraging, but by no means sufficient to establish the suitability of epoxy adhesives for sealing high reliability hybrid microcircuits. Much remains to be done to determine the degree to which epoxy adhesives are suitable for this application, and to establish an adequate data base for writing a guideline specification for the selection and qualification of epoxy adhesives.

The objective of the recommended follow-on study, Phase II, is to continue the experimental effort, culminating it either in the development of a guideline specification or in the rejection of epoxy adhesives for this application. As shown in the Study Flow Plan given as Figure 7, the effort to accomplish the objective of the recommended Phase II study will consist of four tasks. A description of these tasks follows.

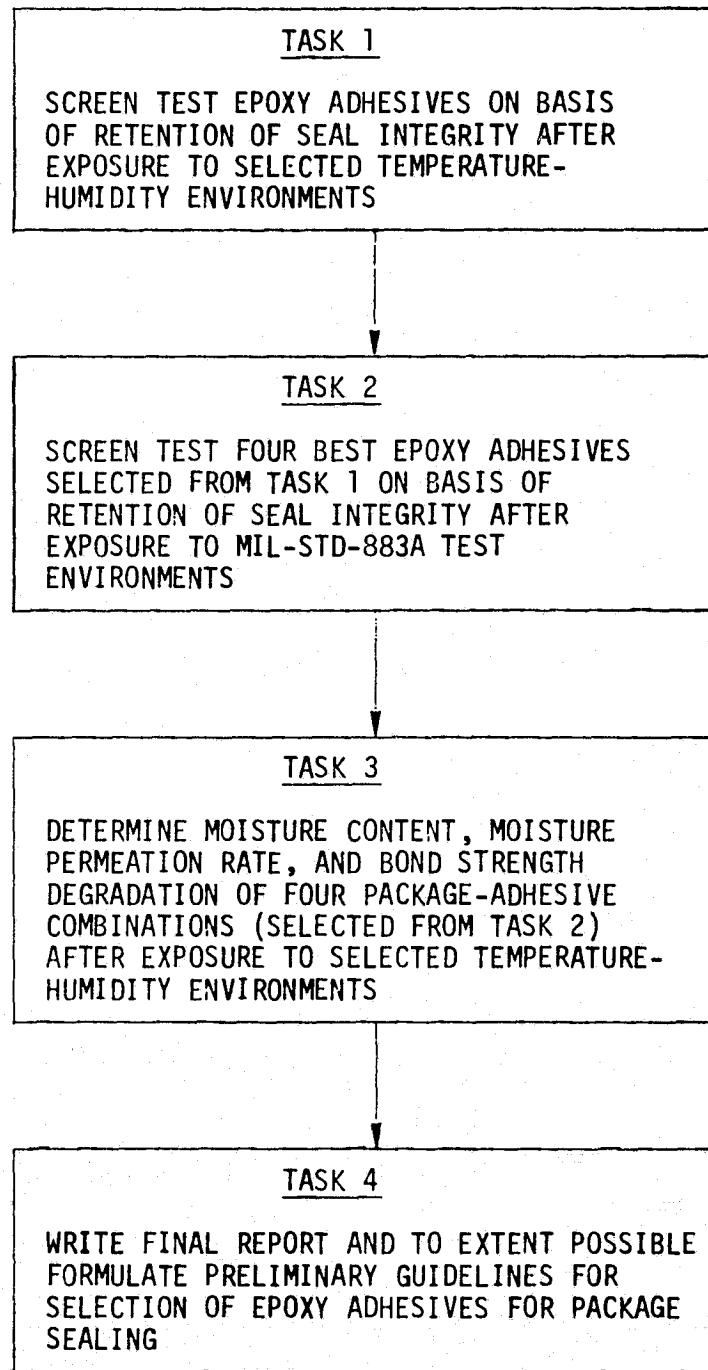


Figure 7. Phase II Study Flow Plan

**TASK 1. Screen Test Epoxy Adhesives on the Basis of Retention of Seal Integrity After Exposure to Increasingly Severe Temperature-Humidity Environments**

- a. Seal a number of 2.54 cm (1 inch) square gold-plated Kovar packages with each of ten epoxy adhesives as required to conduct screening tests.
- b. Seam seal 12 similar packages to serve as controls for screening tests.
- c. Expose the control packages and the epoxy sealed packages as appropriate to 50°C/60% RH, 60°C/98% RH, Ten-day Moisture Test, and 85°C/85% RH environments.
- d. Seal-test packages after such exposure in accordance with MIL-STD-883A, Method 1014.1, Test Conditions A<sub>2</sub> and C<sub>2</sub> to determine seal integrity.
- e. Select four best epoxy adhesives for further testing in Task 2.

**TASK 2. Evaluate Epoxy Adhesives on the Basis of Retention of Seal Integrity After Exposure to MIL-STD-883A Screen Tests**

- a. Seal thirty 2.54 cm (1 inch) square gold-plated Kovar packages and thirty 2.29 cm (0.9 inch) square ceramic packages with each of the four epoxy adhesives selected in Task 1.
- b. Seam seal thirty similar gold-plated Kovar packages to serve as controls for screening tests.
- c. Expose six of the control packages and six of the gold-plated Kovar and ceramic packages sealed with each of the adhesives, individually and sequentially to thermal shock (15 cycles, -65°C to +150°C), temperature cycling (15 cycles, -65°C to +150°C), mechanical shock (5 shocks at 1,500 g's), constant acceleration (5,000 g's), and temperature aging (240 hours at 125°C).

- d. Seal-test packages after such exposure in accordance with MIL-STD-883A, Method 1014.1, Test Conditions A<sub>2</sub> and C<sub>2</sub> to determine seal integrity.
- e. On the basis of these results and those of Task 1, select four package-adhesive combinations for further evaluation in Task 3.

**TASK 3.** Determine Moisture Content, Moisture Permeation Rate, and Degradation of Bond Strength of Epoxy Adhesive Sealed Packages Due to Exposure to Selected Temperature-Humidity Environments

- a. Perform moisture analyses on three packages of each type including seam sealed controls which have not been exposed to any of the temperature-humidity conditions to determine baseline data.
- b. Subject nine packages of each type to each of the following temperature-humidity conditions: 60°C/98% RH, 85°C/50% RH, and 85°C/85% RH.
- c. Remove three of each nine packages from the specified test environments after 24 hours, seal-test them, analyze them for moisture content, and centrifuge them at 5,000 g's to determine if bond strength has been degraded.
- d. Remove the remaining six packages three at a time after additional exposure intervals determined on basis of previous results, and similarly test them.
- e. Correlate and analyze results and calculate moisture permeation rates for tested adhesives.

**TASK 4. Write Final Report**

Assess and collate experimental data from Tasks 1 through 3 and, to the extent possible, formulate preliminary guidelines for selecting epoxy adhesives for package sealing.

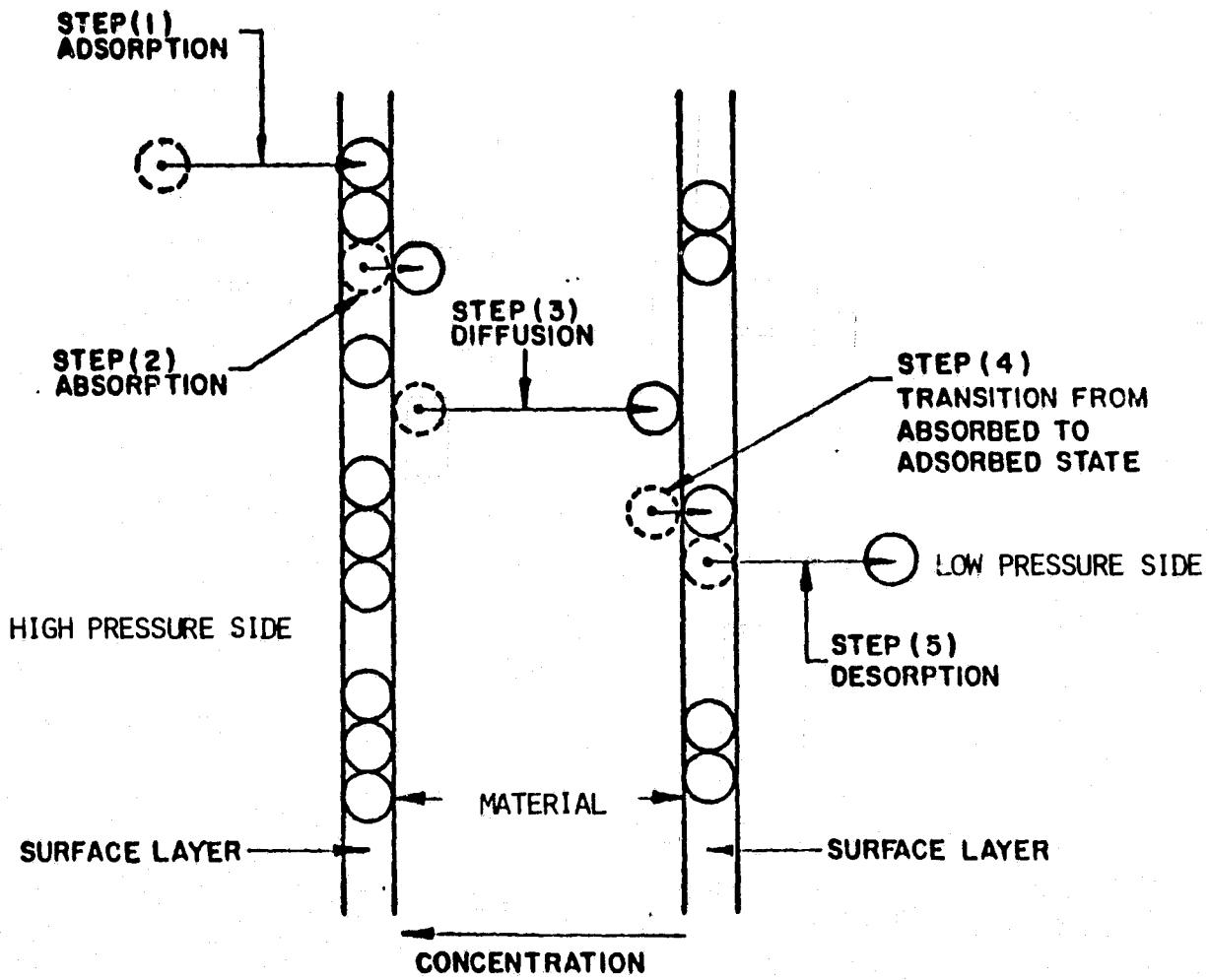
## APPENDIX

## DISCUSSION OF GAS PERMEATION

The fact that low leak rates are obtained when seal integrity is determined by Test Conditions A<sub>1</sub> or A<sub>2</sub> of MIL-STD-883A, Method 1014.1 does not necessarily mean that moisture has not entered the packages. For these tests, helium is used as the tracer gas, and as the following discussion will show, the rates of helium permeation and moisture permeation are not directly correlatable.

The permeation of a gas through a material results from both convection or flow through channels, cracks or flaws of sufficient size in the material and diffusion or direct penetration through the lattice structure of the material. In those cases where permeation is dominantly due to convection, the permeation rates of different gases are correlatable. The different gases will permeate in relative amounts determined by the inverse ratio of the square roots of their molecular masses. In cases where diffusion is the dominant process, this is not so. The diffusion process consists of the following steps as illustrated in the accompanying figure:

1. Adsorption of the gas atoms or molecules on the surface of the material, possibly followed by dissociation.
2. Absorption of the gas by the material to some equilibrium solubility value.
3. Diffusion or movement of the gas atoms or molecules through the interior of the material under a concentration gradient.
4. Transition of the gas from the absorbed or dissolved state to the adsorbed state.
5. Desorption of the gas atoms or molecules, possibly preceded by recombination.



STEPS IN THE DIFFUSION-ASSOCIATED  
PERMEATION PROCESS

Details of the mechanism of this process differ greatly with the properties of the gas and the material, so the permeation rate due to diffusion for one gas-material system is not correlatable with that of another.

The important convection mechanisms for the present case of package sealing are Poiseuille and Knudsen flow. The flow rates, in atm cc/sec are as follows:

$$\text{Poiseuille Flow: } Q = 1.99 \times 10^5 \frac{a^4}{\eta l} (p_2^2 - p_1^2)$$

$$\text{Knudsen Flow: } Q = 3.05 \times 10^4 \frac{a^3}{l} \sqrt{\frac{T}{M}} (p_2 - p_1)$$

where  $a$  and  $l$  are the radius and length, respectively, of the capillary (channel, crack or flaw) in cm,  $\eta$  is the coefficient of viscosity of the gas in gm/cm sec or poise,  $T$  is the absolute temperature in degrees Kelvin,  $M$  is the molecular mass in grams, and  $p_2$  and  $p_1$  are, respectively, the pressure in atmospheres at the high pressure and low pressure sides of the capillary.

For the diffusion mechanism, the permeation rate in cc (NTP)/sec as derived from Fick's law is as follows:

$$J = D_0 S_0 \frac{A}{d} (p_2^n - p_1^n) e^{-(E' + E'')/RT}$$

where  $D_0$  is the diffusion constant in  $\text{cm}^2/\text{sec}$ ,  $S_0$  is the solubility constant,  $A$  is the surface area of the material in  $\text{cm}^2$  and  $d$  is its thickness in cm,  $E'$  and  $E''$ , respectively, are the activation energies for the diffusion and solution processes,  $R$  is the gas constant,  $T$  is the absolute temperature in degrees Kelvin,  $p_2$  and  $p_1$  respectively are the pressure in atmospheres on the high and low pressure sides of the material, and  $n$  is a constant which is 1/2 for gas-metal systems and 1 for gas-glass and gas-polymer systems over a considerable pressure range.

For gas-polymer systems, which is the case of interest for adhesive sealing, the diffusion of gas molecules through the bulk material occurs as

a result of the kinetic energy of the gas molecules and the molecules of the polymer. Those gas molecules possessing energy equal to or greater than the activation energy which has to be supplied to separate the molecular chains of the polymer a sufficient distance will move through the polymer. Larger openings are required for the diffusion of larger gas molecules, so the required activation energy is greater. Also, the required activation energy depends on the nature of the polymer. It is found that the stronger the secondary valences by which the molecules are interlinked, the greater the required activation energy.

The solubility of a gas in a polymer is also dependent on the nature of both the gas and the polymer. Experiment shows that the solubility of a gas in a polymer is greater, the higher the critical temperature or boiling point of the gas. It is also found that the presence of polar groups in a polymer reduces the solubility of a nonpolar gas, and that polar gas molecules will dissolve more readily in a polymer with polar molecules than in a polymer without polar molecules. This is due to the fact that two polar molecules have greater mutual attraction than a polar and a nonpolar molecule, and as a result, the nonpolar molecule is more or less expelled from the polymer.

This subject is thoroughly treated in Autonetics Technical Memorandum 3041-94-1, "The permeation of Gases Through Solids," by K. L. Perkins, December 7, 1959 (Unpublished) and in a paper entitled "Permeability of Polymers to Gases, Vapors and Liquids" by Alexander Lebovits which was published in Modern Plastics in March 1966.